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*fae* INVESTIGATION OF AIRBORNE CHEMICAL CONCENTRATIONS  
IN CERTAIN WORK AREAS OF NAVAL AIR REWORK  
FACILITY AT NORTH ISLAND, CALIFORNIA

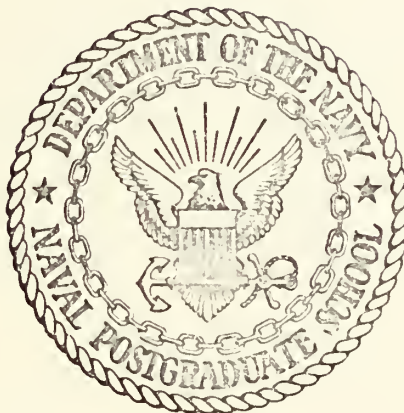
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*ae* AIRCRAFT ENVIRONMENTAL SUPPORT OFFICE  
→ NAVAL AIR SYSTEMS COMMAND  
→ NAVAL AIR REWORK FACILITY  
NORTH ISLAND, CALIFORNIA 92135



# NAVAL POSTGRADUATE SCHOOL

## Monterey, California



INVESTIGATION OF AIRBORNE CHEMICAL CONCENTRATIONS  
IN CERTAIN WORK AREAS OF NAVAL AIR REWORK  
FACILITY AT NORTH ISLAND, CALIFORNIA

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## ABSTRACT

This report covers work done at the Naval Air Rework Facility, North Island (NAVAIREWORKFAC NORIS), California, during October, November, and December 1976. A study of the airborne concentrations of four toxic vapors was made at the Helicopter Blade Bonding Shop and the Electroplating Shop. The four vapors were:

Acetone

Toluene

Methyl Ethyl Ketone (MEK)

Methyl Chloroform

An attempt was made to study a fifth toxic vapor, hydrogen cyanide, but this was unsuccessful because of the inability to calibrate the Wilks Portable Infrared Gas Analyzer (MIRAN I) used in all work done.

Results indicate that, in general, concentrations of the above vapors are within Occupational Safety and Health Act (OSHA) limits for human exposure, based on a long-time (8-hour) average. Occasional peaks were found where limits were exceeded.

The MIRAN I is a useful, dependable vapor concentration analyzer. It should be used to study concentrations of the above vapors and other vapors of possible interest at any part of the North Island complex where humans are exposed to these vapors in the normal course of their work.



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## LIST OF ABBREVIATIONS

AESO	Aircraft Environmental Support Office
LPS	Laboratory Process Specifications
MIRAN I	Wilks Portable Infrared Gas Analyzer
NAVAIREWORKFAC	Naval Air Rework Facility
OSHA	Occupational Safety and Health Act



## I. INTRODUCTION

A. It has been the intention of the Aircraft Environmental Support Office (AESO), Code 64270, North Island, California, to investigate the airborne concentrations of various toxic chemicals used and/or possibly produced in certain operation areas at the North Island facility. In particular, toxic chemicals airborne concentration in two areas were desired:

1. Helicopter Blade Bonding Shop - Shop No. 93432, Bldg. 2
2. Electroplating Facility - Shop No. 7331, Bldg. 472

There have been previous efforts to determine toxic chemical concentrations in these areas. The facility industrial hygienist, Mr. Mike Padilla, has periodically visited the above two areas and made measurements of chemical concentrations. His results have been reported in the Regional Dispensary Report files. In addition, reference (1) reported on evaluation of ambient air in Shop No. 93432, Bldg. 2.

B. The methodology used by Mr. Padilla involved sample collection using air impingers and subsequent wet chemistry work, references (2), (3), and (4), to determine the contents of the impinger solutions. Air impingers work on the principle of bubbling air through various solutions which "capture" by chemical reaction the chemicals whose airborne concentration is desired. The methodology used in reference (1) was a Hydrocarbon Flame Ionization





Detector located in the AESO Mobile Emissions Laboratory. This instrument was not specific for the toxic chemicals tested for acetone and methyl ethyl ketone, but it did, by indirect means, give an indication of the chemical concentration in the ambient shop air. These levels were all well within OSHA eight-hour average exposure limits. The work of Mr. Padilla in the plating area indicated that toxic cyanide levels were within OSHA limits. However, since a specific ion electrode was used to measure impinger collected solutions, the source of the cyanide ion is somewhat obscure. That is, did it come from hydrogen cyanide gas, from cyanide salts that somehow or other became airborne, or from some other source?

C. The time duration of the previous measurements is open to criticism. The work was done infrequently, and when it was done, covered only a small period of daily work. It was thought that a method for monitoring ambient air concentrations as often as desired and for as large a period as desired should be developed. It should be possible to monitor 24 hours a day for several days so as to get a good representative look at shop air contents. Any monitoring method that would cover such periods of time would necessarily require sophisticated instrumentation that would continuously monitor and record concentrations. A suitable instrument was available from the AESO group. It was a Wilks Scientific Corporation MIRAN I Portable Gas Analyzer (No. 5633) with accessories.





1 (A) MIRAN-1 CALIBRATE MODE INCLUDING INJECTION SYRINGE.  
ELECTROPLATE SHOP.



1 (B) MIRAN-SNIFFING MODE ELECTROPLATE SHOP.

FIGURE 1



D. The MIRAN I uses infrared radiation which is passed through (variable path lengths) air containing the material whose concentration is desired. Energy is absorbed by the material and air, and the attenuated radiation (the signal) is detected, measured, and recorded. The MIRAN I is calibrated for a specific material (Appendix A) and is then taken into a shop area to "sniff" the air and record concentrations of the material in question. The concentrations are also recorded on a strip chart recorder for record purposes. The output signal is a voltage in the (0-1) range but is converted and reported in terms of parts per million (ppm). The instrument can remain on station for as long as desired and will give a continuous record of material concentrations that it "sees". The instrument sniffer is a long flexible tube with a cylindrical air inlet at its end. The cylinder contains a filter to protect the instrument optics from particulate matter. The tube is attached to the instrument cell, which in turn is attached to a small air pump, which moves the sample air through the cell; and this provides a continuous sample for monitoring. The instrument with its sniffer tube attached is shown in Figure 1.

E. This report covers the attempted calibration of the MIRAN I for five toxic materials as follows:

1. Acetone
2. Methyl Ethyl Ketone (MEK - also 2 - Butanone)
3. Toluene



4. Methyl Chloroform (1, 1, 1 Trichloroethane)

5. Hydrogen Cyanide (Hydrocyanic Acid) - Calibration

not possible.

The first three of these materials are found in the Blade Bonding Shop and the last two in the Electroplating Shop. It should be noted that hydrogen cyanide is not normally expected to be found in any lethal concentration. However, since tanks of cyanide salts are located in the Electroplating area, it was decided to sniff this area for possible concentrations of hydrogen cyanide if calibration is successful.

F. In addition, the report covers investigations of what materials are used in the two shop areas and, in particular, which of these materials are toxic.

G. Finally, the report presents the results of taking the MIRAN I into the shop areas to monitor the ambient air for the above materials. Due to time limitations, the monitoring covered a period of about one week in each shop.







A. General

1. The writer spent some time in the two previously mentioned shop areas talking to the operating personnel and observing the normal shop operating procedures. It was determined that the materials used in each shop are under the control of a Materials Laboratory Scientist. The people from the Materials Laboratory who currently have responsibility for materials usage in each shop are:

a. Helicopter Blade Bonding Shop - Mr. Jack Diamond, who in turn reports to Ms. Carol Duesler.

b. Electroplating Shop - Mr. Raeburn Solberg, who in turn reports to Mr. Vic Folsom.

2. Some time was spent with Mr. Diamond and Mr. Solberg consulting on how material usage was specified and controlled. The general picture developed is presented in the following material.

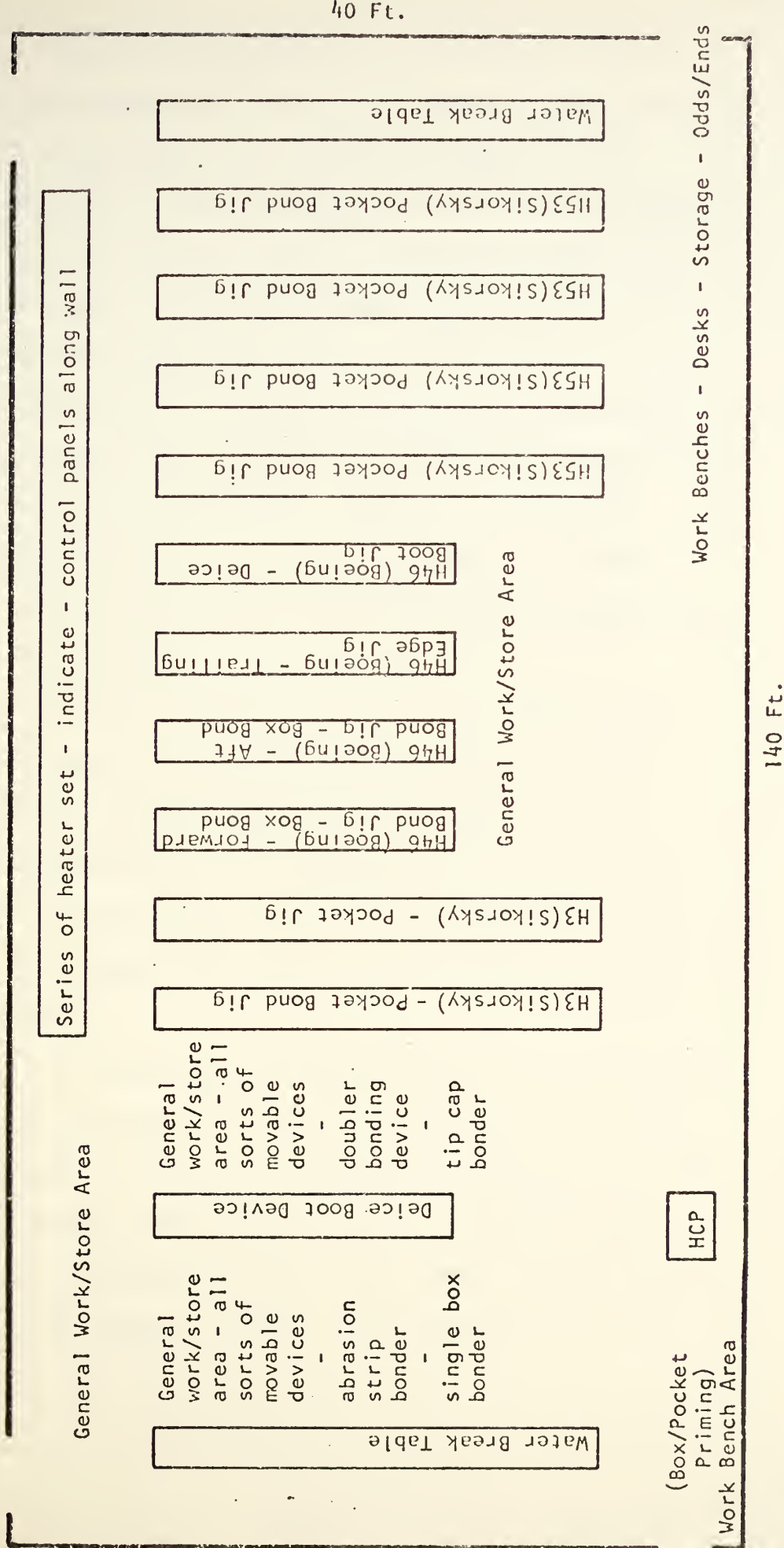
B. Helicopter Blade Bonding Shop

1. A schematic layout of the Helicopter Blade Bonding Shop No. 93432 is shown in Figure 2. All sorts of chemicals are used at various locations in the shop. All materials are checked as received by the Materials Laboratory to insure that specifications are met. Control is maintained via LPS which are on file in the Materials Laboratory and updated as needed. The original source of these LPS's are manuals issued by Boeing and Sikorsky.



FIGURE 2

SCHEMATIC LAYOUT OF BLADE BONDING SHOP NO. 93432, Bldg. 2





2. A list of materials in use is presented in Table I.

This table is not complete, but covers most materials used in any major amount. The solvents are the materials checked in this report. In particular, acetone and MEK were monitored in the shop. The MIRAN I was calibrated for toluene in addition to acetone and MEK. However, during the period the author did this work, toluene was not used in the shop.

3. The Sikorsky H-53 blade has LPS No. 630, addendum 2, 4 August 1975, covering materials used in its cleaning, bonding, etc. The Boeing H-46 blade has LPS No. 630 addendum 1, 6 June 1975, covering materials used in its cleaning, bonding, etc. There is very little H-3 blade work done. The LPS applicable is probably the same as that for the H-53.

4. The main function of the shop is to replace blade boxes/pockets, install trailing edge pieces as needed, install deicing boots, and leading edge abrasive strips, etc. In the process of doing these jobs, it is necessary to strip and clean surfaces and to prepare them for bonding by priming and applying adhesives. The prepared pieces must have heat applied to complete the bonding process.

5. The four solvents in Table I were the ones considered to be toxic and objectionable from the odor standpoint. Of these, only two, acetone and MEK, were investigated in the shop. The other two were not; ethanol because of its small use; toluene because, as noted above, no operations involving it were done during the time of the author's investigation. The four solvents and acetic acid are



TABLE I

## MATERIALS USED IN HELICOPTER BLADE BONDING SHOP NO. 93432, BLDG. 2

Solvents

Acetone  
Methyl Ethyl Ketone (MEK)  
Toluene  
Ethyl Alcohol (Ethanol)

Primers

EC-1290-10% Solids (MMM)  
EC-1660-5% Solids (MMM)  
EC-3917- (MMM)  
EC-3901-12% Solids (MMM)  
EC-2216 A/B  
MMM=Minnesota Mining &  
Mfg. Co. Proprietary  
  
EC-1945

Adhesives

AF-6 Bonding Film (MMM)  
AF-13 Bonding Film (MMM)  
AF-30 Bonding Film (MMM)  
AF-126-2 Bonding Film  
(MMM)  
AF-126-3 Bonding Film  
(MMM)  
AF-6030

Acids

Hydrochloric  
Acetic  
Sulfuric

Cleaners

Cronak (Sodium Dichromate, Sulfuric Acid, Water)  
Cleaning Compound, Aluminum Surface (MIL-C-5410)  
Dalic 1010C  
Turco 2623 (Alkaline Clean)  
Zinc Plate Solution

Degreaser

Methyl Chloroform (1, 1, 1 - Trichloroethane) MIL-T-81533A





normally handled in polyethylene type squeeze bottles with color-coded caps. Color coding is often mixed up by shop personnel. The shop areas where these solvents are used are:

- a. Water Break Table - where blade spar surfaces are cleaned and prepared for bonding.
- b. Work Bench - northeast corner of shop - where boxes/pockets are cleaned, primed, and prepared for bonding.
- c. Various Jigs - where it is occasionally necessary to wipe prepared surfaces with the appropriate solvent. There are other occasional uses of solvent at various other shop locations but usually in small amounts.

6. The primer contents are not exactly known, but they do contain MEK in major amounts. When they are used at the primer work bench or at the jigs, heavy fume concentration is quite noticeable.

7. The bonding process itself involves chemical reaction under heat and pressure. Objectionable fumes are released and are quite noticeable. The chemical content of these fumes is not known. It is suspected that they are a complex mixture of many things among which may be various amines, isocyanates, etc. The MIRAN I may be used to analyze for these chemicals, but the exact fume composition must be known in order to calibrate the instrument. In view of these considerations, no attempt was made to sniff the fumes from the bonding processes.



C. Electroplating Shop

1. A schematic layout of the Electroplating Shop

No. 7331, Bldg. 472, is shown in Figure 3. This layout is based on several engineering drawings obtained through Mr. John Parker, Code 64230. Mr. Parker has been of great help in showing the writer through the electroplating area, explaining the intended functions of the equipment, providing details of construction of the various tanks, and many other ways. Mr. Parker further provided consultation and enlightening discussions during the nine weeks of the writer's residence in San Diego.

2. It should be noted that a number of changes have been made in the use and location of electroplating tanks, that is, changes related to what would be shown on original engineering drawings. Figure 3 indicates the contents of most tanks and indicates the general flow of the sump/drainage system. Shown in particular are the separate drain lines for the cyanide tanks. For the most part, the cyanide drain lines are routed over drainage ditches to the cyanide sump.

3. The materials used in the various tanks are under control of the Materials Laboratory. It is their responsibility to specify what is to go into each tank and the concentration, temperature, degree of agitation purity, pH, etc. This is currently the responsibility of Mr. Raeburn Solberg, who has been of great help in providing the writer with LPS concerning materials used. Mr. Solberg further provided consultation on the makeup, disposal, and general treatment of the various chemical solutions.







4. A list of the various tanks and their chemical contents are shown in Tables II, III, and IV. These tables contain other information of interest such as applicable LPS numbers, tank areas, type of ventilation, OSHA tank classification, and threshold limit values where applicable. Where a material is considered toxic, it is so indicated.

5. The two materials of most interest are cyanide and methyl chloroform. The cyanides are in the form of dissolved salts and normally exist in basic solution (pH 10). The possibility of the existence of hydrogen cyanide over the cyanide tanks or cyanide rinse tanks is of some concern. Since parts are immersed in the various solutions, it is possible that momentary "puffs" of hydrogen cyanide could be generated, if residual acid should contact a cyanide solution or vice versa. The cyanide sump is another possible source of hydrogen cyanide. The possibility of "accidents" or human failure could lead to conditions where appreciable quantities of cyanide solution and acid solution would mix and generate hydrogen cyanide. However, the possibility of this is quite small. Cyanide and cyanide wash-water tanks are constructed with no bottom outlet. They do have overflow weirs to the drain lines and hence to the cyanide sump. Solutions can be totally removed from these tanks by pumping them to bags on trucks, and then removing them to the chemical treatment plant. However, some cyanide solutions have been put in tanks never intended for them, tanks with bottom outlets and with air agitation. Cyanide tanks





TABLE 11-1

## MATERIALS IN ELECTROPLATING TANKS OF LINE P1-SHOP No.7331, BUILDING 472

Tank Number	Function & Lab Process Spec No.	Chemical Solution	Temp. (°F)	Threshold		Tank Surface Area (ft.)	Push Air	Exhaust Air	Required Ventilation Velocity (fpm)	Required Ventilation Volume (cfm)	Toxic
				Limit Value	TLV						
1	Chrome Plate Strip- 294(2A); 298(A3)	Unichrome 80+H <sub>2</sub> O. *	130- 140	2mg/m <sup>3</sup>		18	Yes	Yes	50	1980	No
2	Electro Clean 298(A1)	Turco 4181 *	180- 210	2mg/m <sup>3</sup>		18	Yes	Yes	100	4050	No
3	Ionized H <sub>2</sub> O Rinse	H <sub>2</sub> O	Ambient	-		-	-	-	-	-	-
4	Deoxidizer Acid	50% HCl	Ambient	5 ppm.		18	Yes	Yes	50	1930	No
5	Ionized H <sub>2</sub> O Rinse	H <sub>2</sub> O	Ambient	-		-	-	-	-	-	-
6	Cadmium Strip	NH <sub>4</sub> NO <sub>3</sub>	Ambient	-		18	Yes	Yes	-	-	No
7	Cadmium Plate 261(B); 298(A4)	CdO; NaCN NaOH; H <sub>2</sub> O	Ambient	-		18	Yes	Yes	50	1980	Yes
8	Cadmium Plate	(Same)	Ambient	-		18	Yes	Yes	50	1980	Yes
9	Cadmium Plate	(Same)	Ambient	-		18	Yes	Yes	50	1980	Yes
10	Ionized H <sub>2</sub> O Rinse	H <sub>2</sub> O	Ambient	-		-	-	-	-	-	-
11	DI Rinse	H <sub>2</sub> O	160	-		-	Yes	Yes	-	-	-
12	Drier	Air	160	-		-	-	-	-	-	-

\*Proprietary-Composition Unknown



## MATERIALS IN ELECTROPLATING TANKS OF LINE P2--SHOP No. 7331, BUILDING 472

Tank Number	Function & Lab Process Spec No.	Chemical Solution	Temp. (°F)	Threshold Limit Value (TLV)	Tank Surface Area (ft. <sup>2</sup> )	Push Air	Exhaust Air	Required Ventilation Velocity (fpm)	Required Ventilation Volume (cfm)	Toxic
1	Electro Clean 298(A1)	Oakite 90 (Caustic)*	180-210	2mg/m <sup>3</sup>	18	Yes	Yes	50	1980	No
2	Electro Clean Deruster 298(A1)	Turco 4181* (Caustic)	180-210	2mg/m <sup>3</sup>	18	Yes	Yes	100	4050	No
3	Ionized H <sub>2</sub> O Rinse	H <sub>2</sub> O	Ambient	-	-	-	-	-	-	-
4	Deoxidizer	50% HCl	Ambient	5 ppm.	18	Yes	Yes	50	1980	No
5	1W Rinse-DV Spray	H <sub>2</sub> O	Ambient	-	-	-	-	-	-	-
6	Nickel Strike 261-B; 298(A4)	NiCl HCl H <sub>2</sub> O	Ambient	5 ppm.	18	Yes	Yes	50	1980	No
7	Nickel Plate 277; 298(A4)	Ni Sulfamate 120-Sulfamic Acid 140 Boric Acid H <sub>2</sub> O	120-140	-	-	Yes	Yes	-	-	No
8	Nickel Plate 277; 298(A4)	(Same)	120-140	-	-	Yes	Yes	-	-	No
9	1W Rinse	H <sub>2</sub> O	Ambient	-	-	-	-	-	-	-
10	Electroless Nickel 276; 298(A4)	* (Acid)	190-200	-	18	Yes	Yes	50	1980	No
11	DI Rinse	H <sub>2</sub> O	Ambient	-	-	-	-	-	-	-
12	DI Rinse	H <sub>2</sub> O	160	-	-	-	-	-	-	-
13	Drier	Air	160	-	-	-	-	-	-	-

\*Proprietary-Composition Unknown



Tank Number	Function & Lab Process Spec No.	Chemical Soutlion	Temp. (°F)	Threshold Limit Value (TLV)	Tank Surface Area (ft. <sup>2</sup> )	Push Air	Exhaust Air	Required Ventilation Velocity (fpm)	Required Ventilation Volume (cfm)	Toxic
1	Electro Clean 298(A4)	Oakite 90 (Caustic)* 243	235-243	2mg/m <sup>3</sup>	18	Yes	Yes	100	4050	No
2	IW Rinse	H <sub>2</sub> O	Ambient	-	-	-	-	-	-	-
3	Descale-Deoxidize	HNO <sub>3</sub> -HF	Ambient	5ppm	18	Yes	Yes	50	1980	No
4	IW Rinse	H <sub>2</sub> O	Ambient	-	-	-	-	-	-	-
5	(Not Used)	-	-	-	-	-	-	-	-	-
6	IW Rinse	H <sub>2</sub> O	Ambient	-	-	-	-	-	-	-
7	Reverse Etch	H <sub>2</sub> SO <sub>4</sub>	Ambient	1mg/m <sup>3</sup>	18	Yes	Yes	50	1980	No
8	Nickel Strike 261B; 298(A4)	NiCl <sub>2</sub> HCl	Ambient	5ppm	18	Yes	Yes	-	-	-
9	Nickel Plate	(Same as P2-7)	120-140	-	18	Yes	Yes	50	1980	No
10	IW Rinse-DI Spray	H <sub>2</sub> O	Ambient	-	-	-	-	-	-	-
11	Tin Plate 298(A4)	Na Stannate NaOH Na Acetate Na Perborate	140-160	-	-	Yes	Yes	-	-	-
12	IW Rinse-DI Spray	H <sub>2</sub> O	Ambient	-	-	-	-	-	-	-
13	Cadmium Plate 261B; 298(A4)	(Same as P1-7)	Ambient	-	18	Yes	Yes	50	1980	Yes
14	Rinse	H <sub>2</sub> O	160	-	-	-	-	-	-	-
15	Drier	Air	160	-	-	-	-	-	-	-

\*Proprietary-Composition Unknown



Tank Number	Function & Lab Process Spec No.	Chemical Solution	Temp. (°F)	Threshold Limit Value (TLV)	Tank Surface Area (ft. <sup>2</sup> )	Push Air	Exhaust Air	Required Ventilation Velocity (fpm)	Required Ventilation Volume (cfm)	Toxic
1	Non-etch Clean 281B 298A1	Turco 4080 (Caustic) *	160-180	1mg/m <sup>3</sup>	18	Yes	Yes	100	4050	No
2	IW Rinse	H <sub>2</sub> O	Ambient	-	-	-	-	-	-	-
3	Etch	HNO <sub>3</sub> -HF	Ambient	2ppm	18	Yes	Yes	50	1980	No
4	IW Rinse	H <sub>2</sub> O	Ambient	-	-	-	-	-	-	-
5	Zn-Al COAT (Caustic)	NaCN ZnO <sub>2</sub>	Ambient	1mg/m <sup>3</sup>	18	Yes	Yes	.50	1980	No
6	IW Rinse	H <sub>2</sub> O	Ambient	-	-	-	-	-	-	-
7	Copper Strike 281B; 298A4	CuCN NaCN Rochelle Salt Wetting Agent	140-160	5mg/m <sup>3</sup>	18	Yes	Yes	75	3060	Yes
8	IW Rinse- DI Spray	H <sub>2</sub> O	Ambient	-	-	-	-	-	-	-
9	Nickel Plate 277; 298A4	(Same as P2-7)	120-140	-	18	Yes	Yes	50	1980	No
10	IW Rinse- DI Rinse	H <sub>2</sub> O	Ambient	-	-	-	-	-	-	-
11	Silver Strike 270(1); 298A4	AgCN NaCN	Ambient	-	-	Yes	Yes	-	-	Yes
12	Silver Plating 270(1); 298A4	AgCN KCN K <sub>2</sub> CO <sub>3</sub> - KOH Brightener	Ambient	-	-	Yes	Yes	-	-	Yes
13	IW Rinse	H <sub>2</sub> O	Ambient	-	-	-	-	-	-	-
14	DI Rinse	H <sub>2</sub> O	160	-	-	-	-	-	-	-
15	Drier	Air	160	-	-	-	-	-	-	-

\*Proprietary-Composition Unknown





Tank Number	Function & Lab Process Spec No.	Chemical Solution	Temp. (°F)	Threshold Limit Value TLV	Tank Surface Area (ft. <sup>2</sup> )	Push Air	Exhaust Air	Required Ventilation Velocity (fpm)	Required Ventilation Volume (cfm)	Toxic
1	Electro Clean 298A1	Oakite 90 (Caustic)* 180	160-	2mg/m <sup>3</sup>	18	Yes	Yes	75	3060	No
2	1W Rinse	H <sub>2</sub> O	Ambient	-	-	-	-	-	-	-
3	Deoxidize	HCl HNO <sub>3</sub>	Ambient	-	(Small Insert Tanks)	Yes	Yes	50	1980	No
4	1W Rinse	H <sub>2</sub> O	Ambient	-	-	-	-	-	-	-
5	Copper Strike 281B; 298A4	(Same as P4-7)	140-160	5mg/m <sup>3</sup>	18	Yes	Yes	75	3060	Yes
6	Copper Plate 265; 298A4	CuCN NaCN NaOH Na <sub>2</sub> CO <sub>3</sub>	140-160	5mg/m <sup>3</sup>	18	Yes	Yes	75	3060	Yes
7	1W Rinse	H <sub>2</sub> O	Ambient	-	-	-	-	-	-	-
8	(Tank Empty)	-	-	-	-	-	-	-	-	-
9	(No Tank-Space Open)									
10	Nickel Strip 294(5A)	Enstrip* NaCN	120-140	-	-	Yes	Yes	-	-	Yes
11	Sodium Cyanide (Insert Tank)	NaCN	Ambient	-	-	Yes	Yes	-	-	Yes
12	Cadmium Conversion 257; 298A3	Irridite 1A & 1B* (Acid)	75-90	-	-	Yes	Yes	-	-	No
13	1W Rinse	H <sub>2</sub> O	Ambient	-	-	-	-	-	-	-
14	DI Rinse/Spray	H <sub>2</sub> O	120-140	-	-	-	-	-	-	-
15	Drier	Air	160	-	-	-	-	-	-	-

\*Proprietary-Composition Unknown



Tank Number	Function Lab Process Spec No.	Chemical Solution	Temp. (°F)	Threshold Limit Value TLV	Tank Surface Area (ft. <sup>2</sup> )	Push Air	Exhaust Air	Required Ventilation Velocity (fpm)	Required Ventilation Volume (cfm)	Toxic
1	(Tank No. Longer Exists)		-	-	-	-	-	-	-	-
2	Degrease 113 B	Methyl Chloroform	165	350ppm	50	-	Yes	50	5533	Yes
3	Wax Strip	H <sub>2</sub> O	160	-	-	-	-	-	-	-
4	Chrome Plate 298A4	CrO <sub>3</sub> H <sub>2</sub> SO <sub>4</sub>	128-132	0.5mg/m <sup>3</sup>	32.6	Yes	Yes	75	6194	No
5	(Same)	(Same)	(Same)	(Same)	(Same)	(Same)	(Same)	(Same)	(Same)	No
6	(No Longer Exists)									
7	1W Rinse	H <sub>2</sub> O	Ambient	-	-	-	-	-	-	-
8	Chrome Plate 298A4	(Same as P6-4)								
9	Chrome Plate 298A4	(Same as P6-4)								
10	1W Rinse	H <sub>2</sub> O	Ambient	-	-	-	-	-	-	-
11	Rinse	H <sub>2</sub> O	180°	-	-	-	-	-	-	-
12 } 13 } 14 } 15 }	Chrome Plate	(Same as P6-4)								
Not Used										
16	Self Reg. Chrome	Chrome No. 10	128-132	0.5mg/m <sup>3</sup>	6.25	Yes	Yes	75	1188	No
17	1W Rinse	H <sub>2</sub> O	Ambient	-	-	-	-	-	-	-
18	Derust	Caustic	180-200	2mg/m <sup>3</sup>	6.25	Yes	Yes	100	1400	No
19	(No Longer Exists)									
Special Tank 7N	Masking (Maskoat #2)	Eronel (Polyvinyl Butyrate)	375-385	-	6.4	-	Yes	100	640	Toxics Plus Burn Hazard



Tank Number	Function & Lab Process Spec No.	Chemical Solution	Temp. (°F)	Threshold Limit Value		Tank Surface Area (ft. <sup>2</sup> )	Push Air	Exhaust Air	Required Ventilation Velocity (fpm)	Required Ventilation Volume (cfm)	Toxic
				TLV	TLV						
1	Silver Strip 294(8)	NaCN NaOH	Ambient	2mg/m <sup>3</sup>	(Same)	19.8	Yes	Yes	75	3366	Yes
2	Silver Strip 294(8)	(Same)	(Same)	(Same)	(Same)	(Same)	(Same)	(Same)	(Same)	(Same)	Yes
3	Nickel Strip 294(5A)	Enstrip NaCN	120- 140	5mg/m <sup>3</sup>	(Same)	11.6	Yes	Yes	75	1972	Yes
4	Aluminum Chrome Strip 294(6); 298A3	Chromic- Sulfuric Acid	70- 130	0.5mg/m <sup>3</sup>	(Same)	13.7	Yes	Yes	75	2603	No
5	IW Rinse	H <sub>2</sub> O	Ambient	-	(Same)	-	-	-	-	-	-
6	Special Strips	Chromic- Sulfuric- Nitric Acids	Ambient	2ppm	(Same)	29.1	Yes	Yes	50	2910	Yes
7	Strip	Hcl-CONC	Ambient	5ppm	(Same)	18.6	Yes	Yes	50	2046	No
8	Cold H <sub>2</sub> O Rinse	H <sub>2</sub> O	Ambient	-	(Same)	11.6	-	-	-	-	-
9	Hot H <sub>2</sub> O Rinse	H <sub>2</sub> O	160	-	(Same)	11.6	-	-	-	-	-

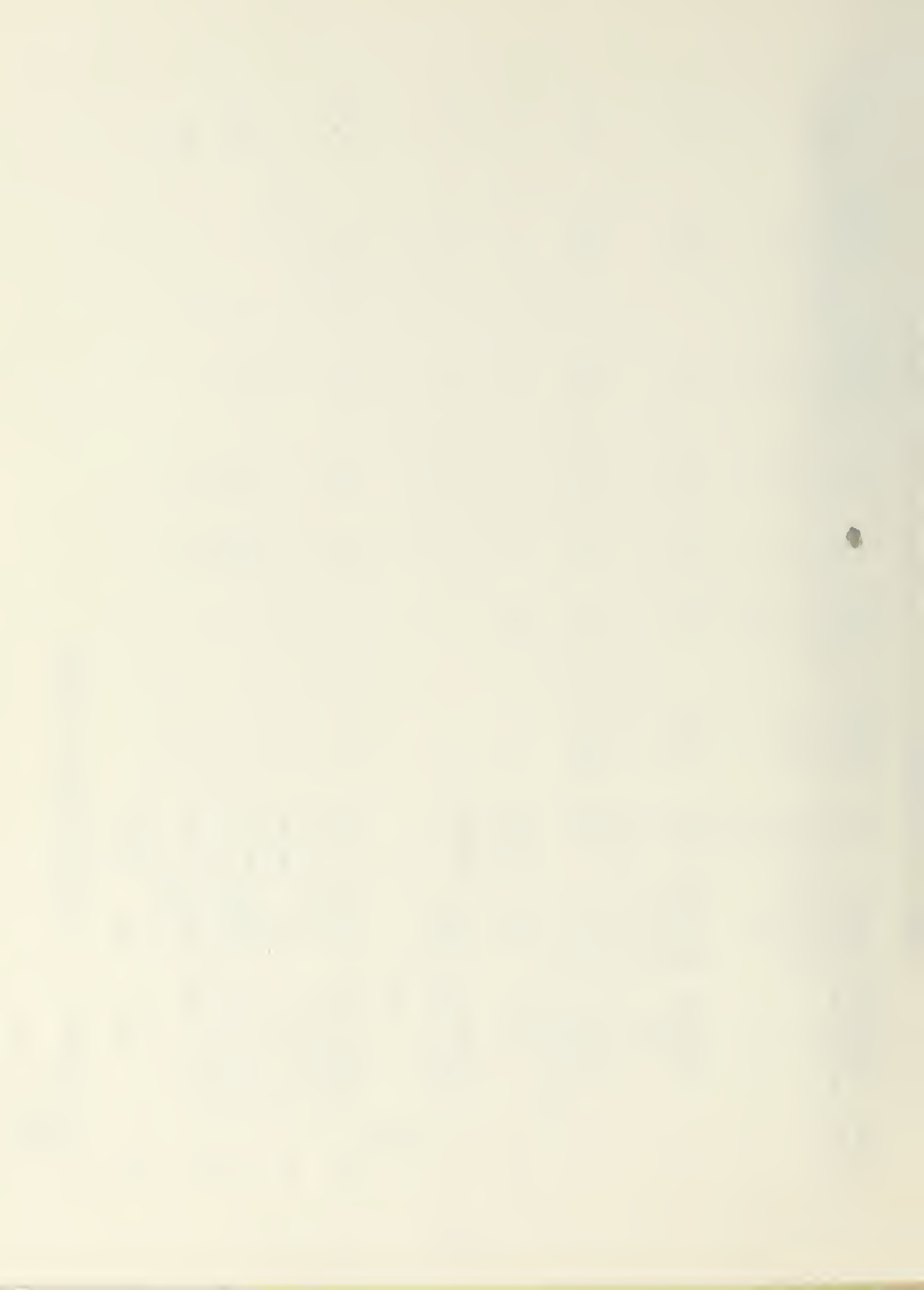


MATERIALS IN ELECTROPLATING TANKS OF LINE P8-SHOP No. 7331, BUILDING 472

Tank Number	Function & Lab Process Spec No.	Chemical Solution	Temp. (°F)	Threshold Limit Value TLV	Tank Surface Area (ft. <sup>2</sup> )	Push Air	Exhaust Air	Required Ventilation Velocity (fpm)	Required Ventilation Volume (cfm)	Toxic
1	Derust	NaOH (Turco 4181)	190-200	5mg/m <sup>3</sup>	26	Yes	Yes	75	4420	No
2	DI Rinse	H <sub>2</sub> O	140-160	-	26	-	-	-	-	-
3	Phosphate Coat-222A 298A3	Parcolube* Phosphoric Acid, Fe	205-210	0.1mg/m <sup>3</sup>	11.6	Yes	Yes	75	2000	No
4	Derust	(Same as 1 above)								
5	Oxide on Steel 224; 298A1	* (Caustic)	295-297	2mg/m <sup>3</sup>	26	Yes	Yes	100	5200	No
6	Rinse	H <sub>2</sub> O	160-180	-	26	-	-	-	-	-
7	(Special Small Tank)	Chromic Acid	Ambient	-	-	-	-	-	-	-
8	Do Not Exist									
9										
10	Nickel Plate 277; 298A4	(Same as P2-7)	120-140	-	-	Yes	Yes	-	-	No
11	Rinse	H <sub>2</sub> O	Ambient	-	-	-	-	-	-	-
12	Silver Plate	(Same as P4-12)	Ambient	-	-	Yes	Yes	-	-	Yes
13	Cadmium Plate	(Same as P1-7)	Ambient	-	-	Yes	Yes	-	-	Yes
14	Rinse	H <sub>2</sub> O	Ambient	-	-	-	-	-	-	-

\*Proprietary-Composition Unknown

† Series of Special Small Tanks Known as "The Barrel Line"





should have no air agitation. Also, it is occasionally necessary to recirculate solutions using portable pumps and lines. In view of all this plus the possible "act-of-God" type of failure, such as earthquake and massive tank rupture, there is always a small but infinite possibility of large amounts of cyanide solution contacting a large amount of acid and generating hydrogen cyanide.

6. The writer's main interest was to use the MIRAN I to sniff for hydrogen cyanide at any point where it might exist. The MIRAN I might be calibrated for hydrogen cyanide and could possibly provide a sensitive way of detecting this material. Also, since there is a degreasing tank in the Electroplating Shop, the MIRAN I was calibrated for methyl chloroform and then used to sniff the air over and around this tank.

7. It should be noted that the MIRAN I could be used to sniff for other materials in this area such as various acids, etc., but it was not the purpose of this work to do material other than cyanides and chloroform. There is one bothersome material which may be amenable to infrared detection. This is the hot mask material, Mask Coat No. 2, also called Eronel, which is classed as both a toxic and burn hazard (used at about 400°F). The tank containing this material has air suction venting along one edge, but it still gives off a considerable objectionable odor. The chemical makeup of the material is not known. It is suspected to contain a polyvinyl



butyrate, plus other chemicals. To sniff this tank with the MIRAN I would require a knowledge of the chemical vapors given off so calibration work could be done. However, there is some worry about doing this because of possible vapor condensation on instrument optics with resulting ruin of the instrument. It is the writer's understanding that serious consideration is being given to replacing the Mask Coat No. 2 with ordinary wax. If this is done, potential toxicity/burn problems would disappear.



### III. CALIBRATION OF THE MIRAN I INFRARED PORTABLE GAS ANALYZER

#### A. General

1. A complete description of the MIRAN I portable infrared gas analyzer is given in Appendix A. Section 3 on operations contains the calibration procedure. This procedure was followed exactly for the five materials dealt with in this report.

2. Small amounts of relatively pure:

Acetone	- Liquid - Boil Point	56.5°C
Methyl Ethyl Ketone	- Liquid - Boil Point	79.6°C
Toluene	- Liquid - Boil Point	110.6°C
Methyl Chloroform	- Liquid - Boil Point	74.1°C
Hydrogen Cyanide (HCN)	- Gas - Boil Point	26.0°C

were obtained. The liquid forms were injected into the calibration system using hypodermic syringes. Hydrogen Cynaide was injected using a gas-tight syringe. Since microliter ( $\mu$ l) amounts were used, the liquids vaporized very quickly so that an equilibrium concentration was rapidly established.

3. Initial checkout procedures and preliminary steps of Section 3 were done and results recorded in the record book, which is really Appendix B. Since this record book is the repository of permanent records of work done for this report, copies of specific things alluded to will be made. There is, of course, only one copy of the record book. Initial checkout involved generating the 2.5 to



14.5 micron ( $\mu$ ) wavelength sweep for air so it could be compared to what the MIRAN I instruction book said it should look like. This was done and the results are shown in Figure 4. It should be compared to Figure 3-1 of Appendix A. Figure 4 is a copy of Page 11 of the record book. It can be seen that the form of the generated sweep agrees quite well with the typical curve. The mode of operation during the cure generation is that of the sniffing mode shown in Figure 3-2 of Appendix A. Everything appeared normal, so calibration of the above five material proceeded. Pertinent parameters for these material are indicated in Table V. Typical spectra for air and other materials are available in reference (5), (6), (7) and (8).

4. The data of Table V served as orienting data.

The path length and wavelength indicated would be selected; but all possible parameters would be varied to try to maximize the signal produced, in order to get as much scale reading as possible. This involved varying the gain, slit opening, and path length. The wavelength was varied slightly around the recommended wavelength. This was necessary because of the slight amount of "play" in the wavelength selection wheel. The time constant was also varied so as to give a relatively fast signal response.

B. Calibration Procedure

1. The procedure will be outlined. See the record book for details.





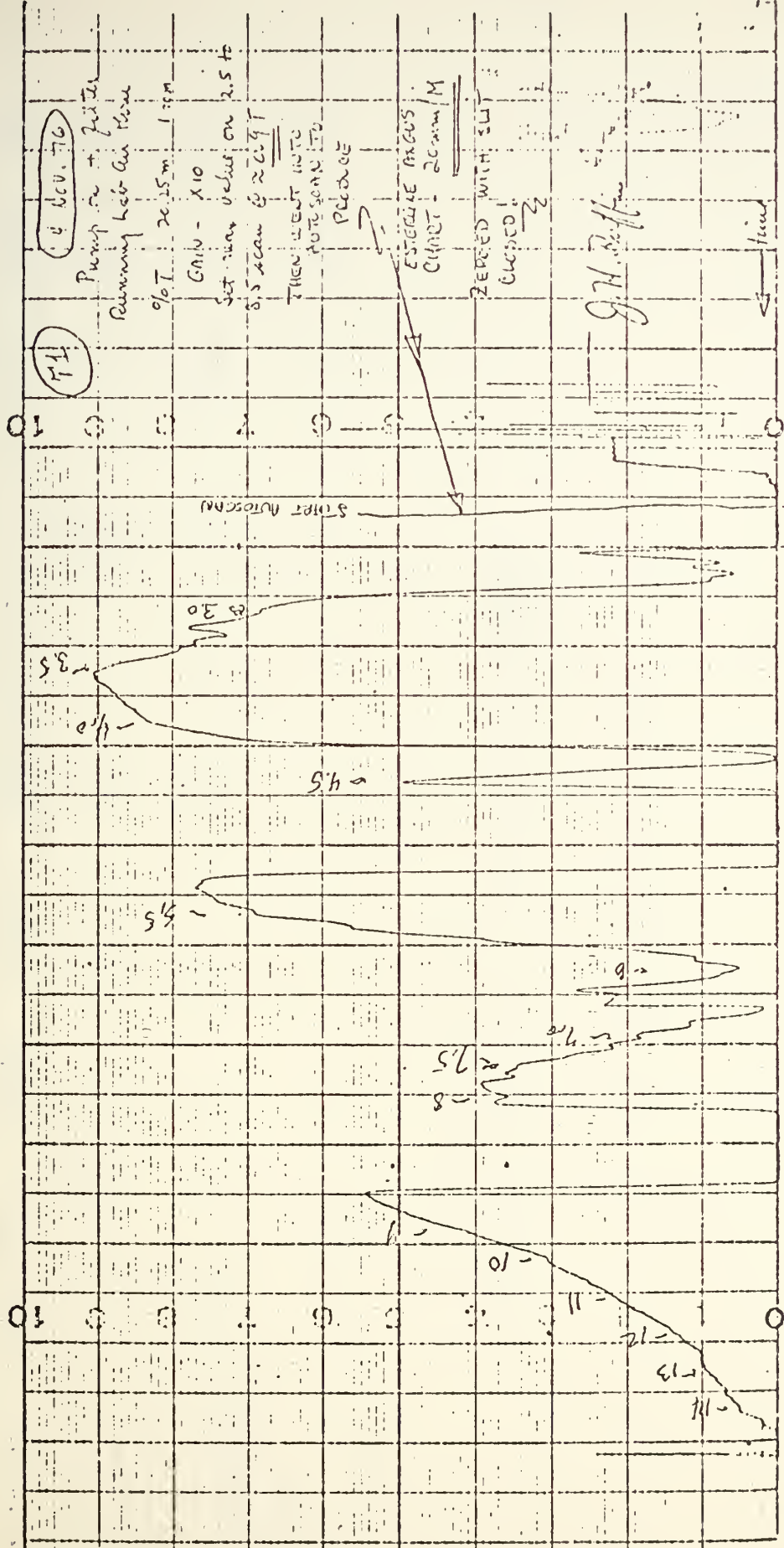


FIGURE 4

MIRA 1 Output When Air Is Scanned From 2.5 to 14.5 MICRONS  
At Conditions Indicated



TABLE V

## CALIBRATION PARAMETERS FOR FIVE MATERIALS CALIBRATED FOR MIRAN I WORK

Material	Max Allowable Exposure (8 Hr.) PPM	Analytical Wavelength Microns (m)	Min Concentration Detectable PPM	Absorbance To OSHA Limit	Scale Exp	Path Length (m)	Molecular Weight	Density gm/ml (20/4)	Boil Point (°C)
Acetone	10 <sup>3</sup>	2.4.10 <sup>3</sup>	8.2	0.09	0.49	1	58.08	0.792	56.5
Methyl Ethyl Ketone (2-butanone)	0.2.10 <sup>3</sup>	0.59.10 <sup>3</sup>	8.5	0.2	0.52	1	72.1	0.805	79.6
Toluene	0.2.10 <sup>3</sup>	-	13.7	0.5	0.38	1	92.1	0.867	110.6
Methyl Chloroform (1, 1, 1 Trichloroethane)	0.35.10 <sup>3</sup>	1.9.10 <sup>3</sup>	13.9	0.05	0.56	1	133.4	1.325	74.1
Hydrogen Cyanide (HCN)	10 (Skin)	11 14.1†	1	*	*	*	27.0	0.688	26.0

(Skin)

† Available from work done in reference (b).

\* Not available from MIRAN tables. Will have to establish by trial and error.



a. The first step was to estimate the number of microliters of liquid or gas needed to produce concentrations up to and somewhat beyond those of maximum allowable exposure. The volume of the cell and recirculating pump and lines was known to be 5.64 liters. All work was done at room temperature (297°K) and atmospheric pressure of (1 atm). Based on this the following equation was used:

$$V_{LIQ.} (\mu l) = \frac{(C_{ppm} \cdot MOL WT \cdot P_{ATM} \cdot 5.64)}{LIQ. \cdot R \cdot T \cdot 10^3}$$

where:

$V_{LIQ.}$  = Liquid volume in microliters

$C_{ppm}$  = Concentration in parts per million

$P_{ATM}$  = Pressure in atmospheres (1 atm)

$LIQ.$  = Liquid density

$R$  = Gas constant (0.08205)

$T$  = Absolute temperature (297°K)

Tables were made of  $V(\mu l)$  versus  $C_{ppm}$ . These could be used to relabel the absorbance scale divisions of the recording meter in ppm.

2. Once the approximate liquid quantities were known, the MIRAN I was set up in the calibration mode shown in Figure 3-2 of Appendix A. An amount of pure liquid was prepared in a microliter syringe and injected through the rubber septum of the recirculating pump. The amount used was approximately that corresponding to maximum allowable concentration. When equilibrium



was established, MIRAN I parameters were varied to try to get a signal whose magnitude was at least half scale, if possible. Once the parameters were set, they were maintained for repeated recalibrations so as to obtain repeatable data. Between each calibration, clean air was pumped through the system to purge it. This latter was done in the sniffing mode.

3. Typical results of varying the path length vernier when calibrating acetone are shown in Figure 5. It was immediately obvious that the path length maximums do not occur at exact digits of the vernier dial, but at points in between. The maximum signal is found by roughly setting the vernier, and consequently the path length, to a given value and then varying around this value, usually up scale, to obtain the best signal. Other parameters are varied at the same time to get the best signal possible.

4. With all parameters determined, the cell is purged and a calibration started. Now, however, the liquid is injected one microliter at a time and equilibrium established. This is repeated to 10  $\mu$ l, which is the limit of the liquid syringe. In the case of acetone, a total of 20  $\mu$ l was injected per calibration. A typical curve for acetone is shown in Figure 6.

5. Several consistent calibrations were averaged and the data fitted with a second order polynomial in the least squares sense. This was done on the Wang 2200C, located in Module 1 and belonging to the AESO group.





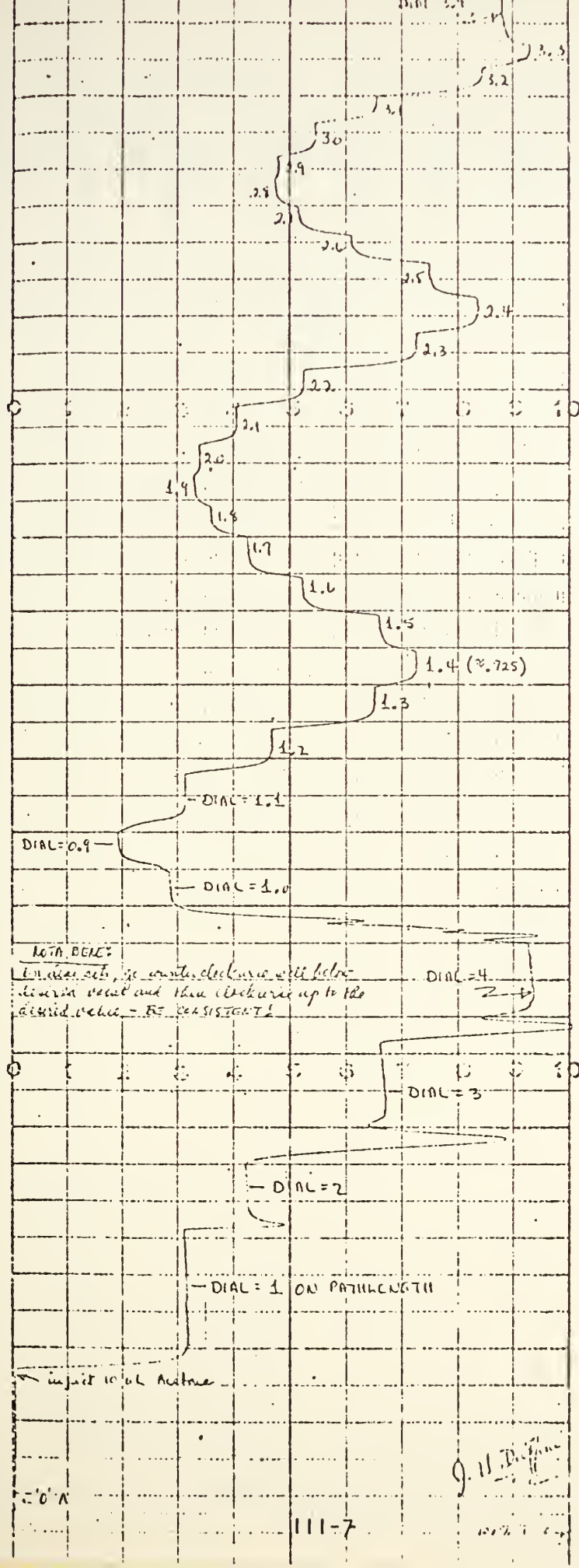


FIGURE 5

MIRAN I Output-10 Microliters of Acetone-Varying Pathlength Vernier

NOTE: BECAUSE the output of the vernier is not linear, it is necessary to use the vernier in the range of 0 to 10 microliters of acetone.

DIAL = 4

DIAL = 3

DIAL = 2

DIAL = 1 ON PATHLENGTH

Input 10.00 Acetone

9.11.11







6. This procedure was followed for each of the materials of Table V. The calibration equations are summarized in Table VI. The calibration parameters actually used are shown in Table VII. A typical calibration curve is shown in Figure 7.

7. The HCN gas needed for attempted calibration was obtained from Fumico, Inc., Amarillo, Texas, in a lecture bottle size (200  $\mu$ l). The gas was released from the slightly warmed bottle ( $\approx 35$  F) by withdrawing it into a gas-tight syringe. The syringe was then used to inject the gas through the rubber septum of the circulating pump of the calibration system. The range of concentration desired was 0-15 ppm. Since the calibration volume is 5.64 liters, an injection of 5.64 microliter ( $\mu$ l) would give a concentration of one ppm. Thus, 15 ppm requires 84.6  $\mu$ l so that a 100  $\mu$ l syringe was used.

8. A literature search indicated the HCN had a fairly strong absorbance peak at 14.1 microns (reference (5)). Initial instrument parameters were:

Slit - 2mm

GAIN - X10

DIAL - 1.4 (= 1 meter)

TIME CONSTANT - 2.5 seconds

and variations were made from these values.

9. Repeated injections of pure HCN gas in the amount of 100  $\mu$ l were made with no response from the instrument. All sorts of parameter variations were tried. Path lengths were varied from 2 to



TABLE VI

## CALIBRATION EQUATION - 2ND ORDER POLYNOMIALS FIT IN LEAST SQUARE SENSE

Acetone: Average of best three sets of data

$$C_{\text{ppm}} = 18.557 + 1731.083A + 1017.483A^2 \quad A = \text{Absorbance Reading} \quad (\text{Max curve error } 3\%)$$

Methyl Ethyl Ketone: Average of best three sets of data

$$C_{\text{ppm}} = 0.931 + 212.283A + 103.166A^2 \quad (\text{Max curve error } 2\%)$$

Toluene: Average of best three sets of data

$$C_{\text{ppm}} = -0.762 + 343.191A + 160.534A^2 \quad (\text{Max curve error } 2.5\%)$$

Methyl Chloroform: Average of best three sets of data

$$C_{\text{ppm}} = 3.142 + 272.361A + 211.513A^2 \quad (\text{Max curve error } 4\%)$$

Hydrogen Cyanide: Could not calibrate.





TABLE VII

## CALIBRATION PARAMETER SETTINGS - MIRAN I

	<u>Acetone</u>	<u>Methyl Ethyl Ketone</u>	<u>Toluene</u>	<u>Methyl Chloroform</u>	<u>Hydrogen Cyanide**</u>
Slit (mm)	1	1	2	1	Varied
Gain	X10	X10	X10	X10	X10
Wavelength (m)	8.1	8.4	13.67	13.75	Varied around 14.1
*Vernier Dial	1.4	13.02	5.91	1.03	Varied
Time Constant (Sec)	2.5	2.5	2.5	2.5	2.5
Chart Speed (mm/M)	20.0	20.0	20.0	20.0	20.0
Max Amount Injected (μl)	20.0	6.0	10.0	10.0	10.0 Gas 2.0 LIQ.

\* These settings always obtained by approaching from the low side.  
This was necessary because of "play" in vernier set.

\*\* No response for HCN for any parameter combination used for amounts of LIQ. and gas indicated.



# METHYL CHLOROFORM CALIBRATION

WAVE 11<sup>th</sup> (-2) ORDER LEAST SQ. DATA FIT

$$C_{ppm} = \frac{3.14701}{1} + \frac{272.3617}{1} A + \frac{211.51252}{1} A^2$$

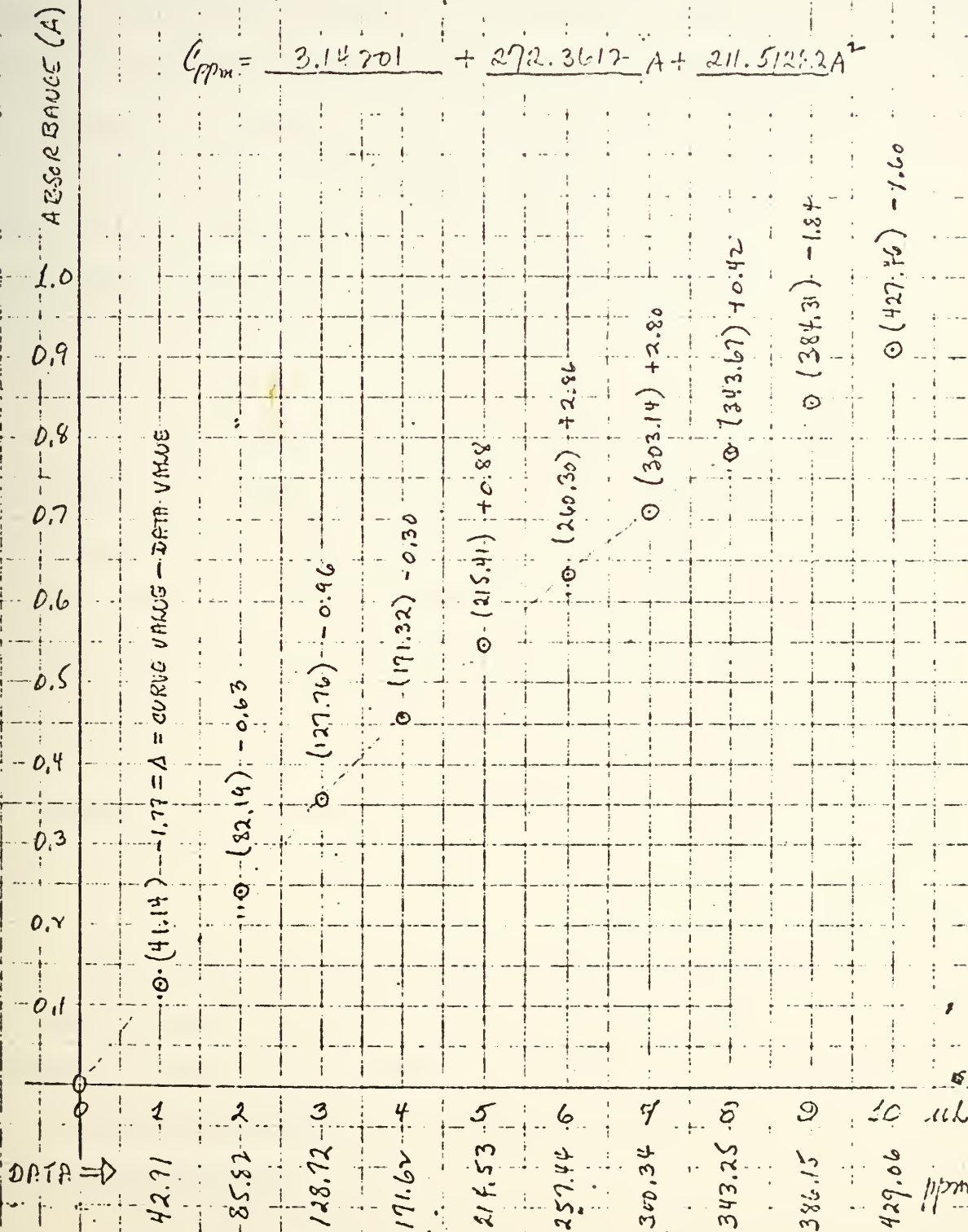


FIGURE 7

Typical Calibration Curve Methyl Chloroform



20 meters, wavelength was varied around  $14.1\mu$  and various slit openings tried. Nothing worked.

10. At this point it was thought that perhaps HCN gas was not actually reaching the instrument. To be absolutely sure that HCN gas was indeed being injected, it was decided to inject liquid HCN. Calculations were made as to the liquid equivalent of  $100\mu\text{l}$  of HCN gas. The figure was  $1.75\mu\text{l}$  of liquid. Accordingly, a liquid syringe was used to inject about  $2\mu\text{l}$  of liquid HCN. There was absolute visual evidence of liquid HCN being injected into the calibration pump. Still there was no instrument response.

11. Further consideration led to a suspicion that the HCN was not staying in the vapor state inside the cell, pump and so forth of the calibration system. It might be condensing on cold metal parts. However, this did not seem too plausible an argument, since higher boiling materials had been successfully calibrated. However to prove the point, the whole system was put in an oven for some time to bring its temperature above the boil point of HCN. Then liquid HCN was injected into the hot instrument. Still no response.

12. Much thought and discussion ensued, with the result being that the reason no signal was obtained was the very low concentration of HCN. The OSHA upper limit for HCN vapors is 10 ppm. This is apparently below the lower detectability limit of the instrument for HCN vapors!



13. To be sure that the MIRAN I was functioning properly, an acetone calibration was done immediately after the last attempted HCN calibration. The instrument worked perfectly on the acetone. At this point any further calibration attempts with HCN were abandoned.





IV. BLADE BONDING/ELECTROPLATING SHOPS AMBIENT AIR MONITORING USING  
THE MIRAN I - RESULTS AND DISCUSSION

A. Helicopter Blade Bonding Shop

1. The MIRAN I was taken to the Helicopter Blade Bonding Shop to sniff for acetone and MEK. Before use on the site, the MIRAN I was checked calibrated for each material. This was done by setting all the parameters as per the calibration work done in Module 1. Due to the slight "play" in the wavelength set wheel, it was necessary to make very small movements with this wheel to get the meter readings to agree with the previous calibration. Once this was done, the MIRAN I was ready for use.

2. The main problem at this point was to be sure of what work was going on where, and at what time, so that the sniffer could be set-up to monitor the work. This involved getting the cooperation of the day shift foreman, Ms. Billy Nelson, and her assistants and workers. They were very interested in what was being done and cooperated fully.

3. The first point at which monitoring was done was the priming work bench (see Figure 2) in the northeast corner. The sniffer was set at about "nose" level and left in place approximately 24 hours. The chart speed of the recorder was slowed to 180mm per hour so as to limit the amount of paper used. There were only a couple of times during this period when box/pocket



cleaning was done using acetone. After cleaning, the box/pocket would be primed, usually with box/pocket 1660 primer. This primer has a lot of MEK in it, yet no signal appeared since the instrument was set specifically for acetone. There were also times during this period when people "played" by soaking either cheesecloth or a cotton wad with acetone and waved it around the sniffer. The result was to drive the pen off scale almost instantaneously. There was also a tendency on the part of personnel to leave the soaked cotton wads and cheesecloth lying around on the bench. This would give rise to higher level readings.

4. The 24-hour period gave too long a record to include in this report. What is shown in Figure 8 is a section of this period which includes box/pocket cleaning and priming and time when no work was done. It is quite obvious that the only time the OSHA limit was exceeded was when people "played". The long-time average exposure would be quite low, less than 20 ppm. The complete 24-hour monitor record is on pages 75 and 77 of the record book (Appendix B).

5. Next, the MIRAN I was next field calibrated and set for MEK. Following this, three primers were sniffed right over the open containers. The primers were 1290, 1660, and 3917. Every one of the primers sniffed gave an off-scale signal, which indicates a high content of MEK. Also sniffed at this time was a waste container into which was thrown cotton and cheesecloth that had been MEK soaked. Since MEK vapor is heavier than air, there is a level of sniffing effect. About one foot above the container the



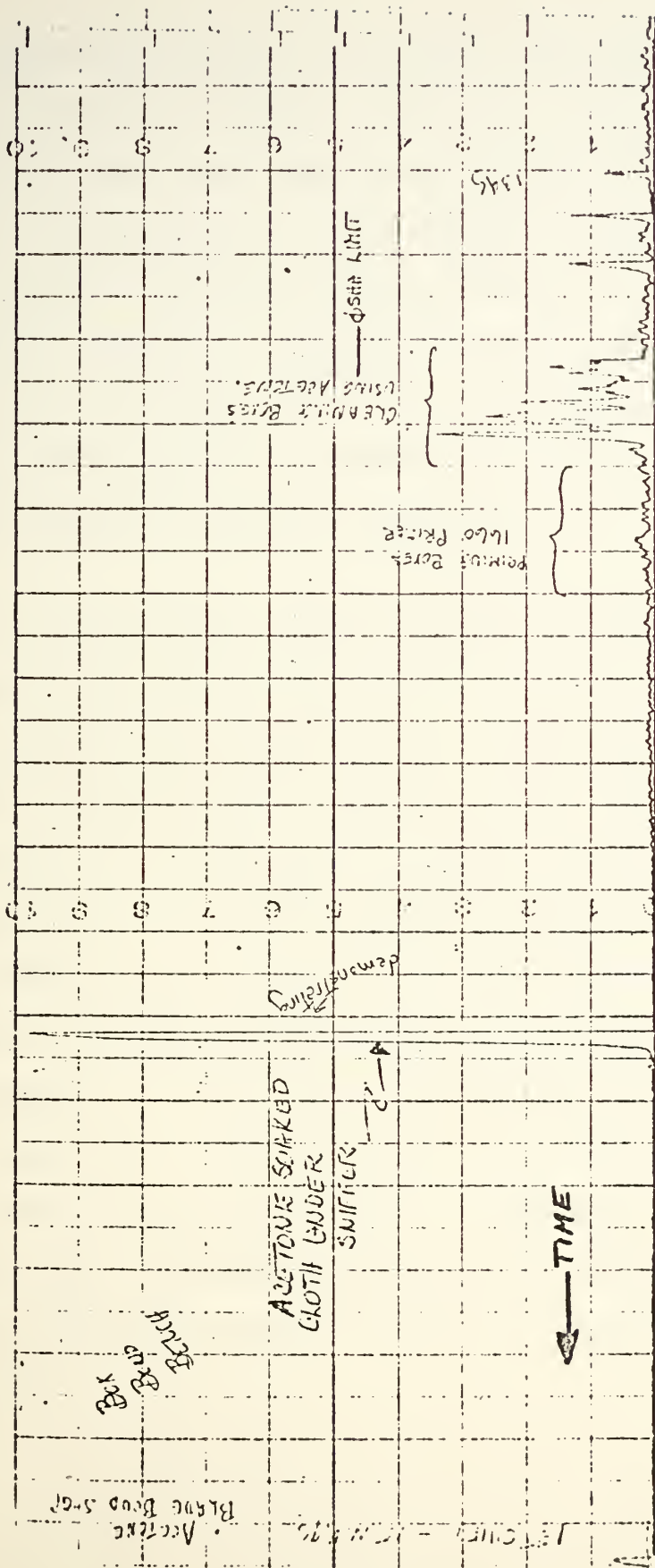


FIGURE 8  
Portion Of Monitoring Of Box/Pocket Bonding Bench  
In Helicopter Blade Bonding Shop



signal is within OSHA limit; but at the top of the container, and slightly below the top, the signal is off scale. Next some cleaning work on the water break table, which is at the east end of the shop was monitored. The cleaning is done by soaking cheesecloth in MEK and wiping the spar at the point where the box/pocket is to be bonded. The MEK comes from polyethylene wash bottles or a gallon jug, usually the wash bottle. The sniffer was located right over the cleaned surface and also at approximate nose position of the worker. These results are shown in Figure 9.

6. At this point, two blades located in jigs in the center of the shop were to be MEK wiped and primed with 3917 primer. A typical "path" through the shop is shown in Appendix C. Noted on this are points at which sniffing was done. The jigs have record books that remain permanently with them. An H-3 blade with some missing pockets was on Jig No. 4250 and another H-3 blade with all pockets missing was on adjacent Jig No. 4249. Both jigs were located about the center of the shop. The record of what occurred during the MEK wipe on Jig No. 4249 and the priming on Jig No. 4250 is shown in Figure 10. It is quite obvious that there are short periods of time when the worker "sees" MEK concentration greater than the OSHA limit. The MEK wiping operation involved carrying a plastic gallon jug of MEK around and using it to soak cheesecloth used for wiping.





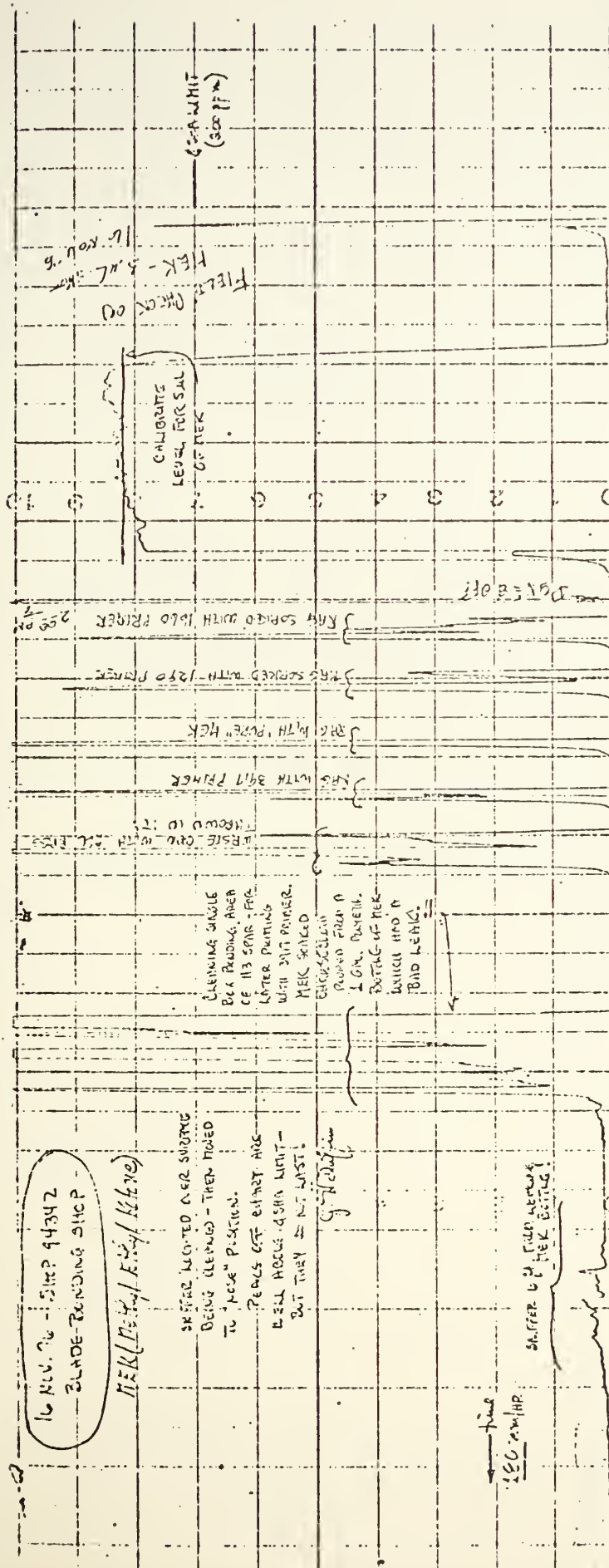


FIGURE 9

Primer Sniffing And Water Break Table Work Sniffing  
Helicopter Blade Bonding Shop



IV-6

FIGURE 10



7. The plastic jug had a leak and MEK pools were left wherever the jug was put down. Also, the soaked cloths were left lying around in a random manner. Thus, the MEK odor was quite noticeable. What one might see from such cloths is shown in Figure 11. The sniffer was located near and far away from a previously soaked cloth lying on a jig.

8. One final piece of work in the Blade Bonding Shop was to recalibrate the MIRAN I for acetone and move the instrument back to the box/pocket priming bench. Then an acetone wipe of some 12 pockets was monitored. The general acetone level during this period was well within the OSHA limit. A couple of peaks approached the limit, but the time-average concentration was quite acceptable. These results are shown on Page 85 of the record book (Appendix B).

#### B. Discussion - Helicopter Blade Bonding Shop Results

1. It should be noted that the work done here was done just after the completion of the installation of a new air conditioning/air movement system for this shop in Building 2. The contractor was having some start-up difficulties with controlling the temperature and humidity in the shop, but in general things were on time as specified. The temperature in the shop should be in the 70-85°F range, and the relative humidity 60%.

2. Prior to the new installation, there apparently were some serious fume problems. The writer did not observe these prior



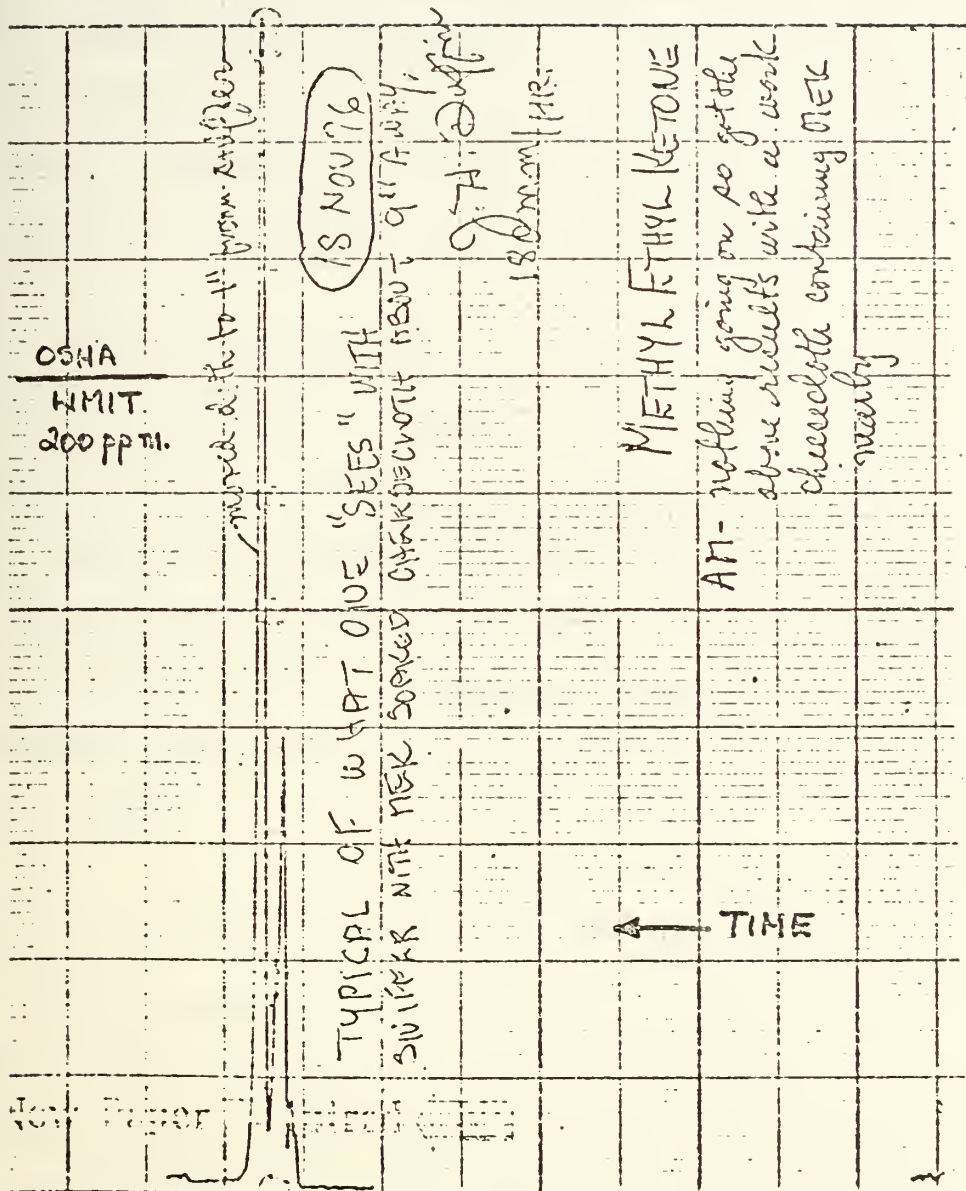


FIGURE 11

Sniffing A Previously MEK Soaked Cheesecloth Discarded On A Jig  
Helicopter Blade Bonding Shop





conditions, but was apprised of the poor condition by the various shop people. It appears that the new air system is a definite improvement over the old system. The only problem of fumes that occurred during the week the writer spent monitoring with the MIRAN I was when the contractor turned on, for the first time, two high power heaters in the air system. Due to "bake out", a noticeable unpleasant odor was present for the better part of the day. This will disappear with time, and so was a "one-time-only" event. However, there were heavy personnel complaints of sore throats, headaches, and so forth, while the odor persisted.

3. The monitoring results for acetone and MEK indicate that acceptable levels of these materials are what normally occur. There will be occasions when peaks of concentration are above the OSHA limit, but they do not persist. There is a close correlation between peaks and material handling by individual workers. Soaking of cotton wads and cheesecloth is common, and then these are left lying around in a random manner. This appears to be the normal way for people to work.

4. Monitoring for longer periods of time could be done to get a more representative record of "normal" shop operations. There might be high activity periods where airborne material concentrations would be higher than those recorded by the writer. However, it would appear longer monitoring periods would not reveal appreciably higher concentration levels. This is purely conjecture on the part of the writer.



5. The MIRAN I proved to be an accurate, sensitive, and dependable instrument for monitoring airborne concentrations of materials used in the shop. The MIRAN I was calibrated for toluene, but no toluene using operations were done during the week the writer spent in the shop. The MIRAN I could be calibrated for other materials such as ethanol (ethyl alcohol), acetic and other acids, amines, and so forth, and then used to monitor for these materials.

6. There was some discussion with shop personnel regarding fumes released during the bonding process where heat and pressure are applied to the primed parts with their accompanying adhesives. The bonding reaction is a complex one, and it would be necessary to determine the composition of the vapors. With the composition known, pure materials could be obtained, insofar as possible, and the MIRAN I calibrated. Then it could be used to sniff and monitor the bonding process.

### C. Electroplating Shop

1. After the Blade Bonding Shop work, the MIRAN I was brought back to Module 1. An air run was done to recheck the instrument and all was in order. Next a check calibration was done on methyl chloroform, and then the instrument was moved to the Electroplating Shop and set up near the degrease tank P7-2 (see Figure 3). It was set on a portable cart so that it could be moved about the shop. The workers use the tank by raising and lowering a basket with a hanging controller located along the east edge of the



tank. The tank itself contains methyl chloroform vapors produced by heating coils at the bottom of the tank. The rising vapors are trapped in the tank by refrigeration coils around the internal periphery, at a point starting 22 inches below the tank top. The rising vapors condense and drip back into the tank. Locating the sniffer at various positions over the open tank should give some idea of the efficiency of the refrigeration coils in containing the vapors within the tank.

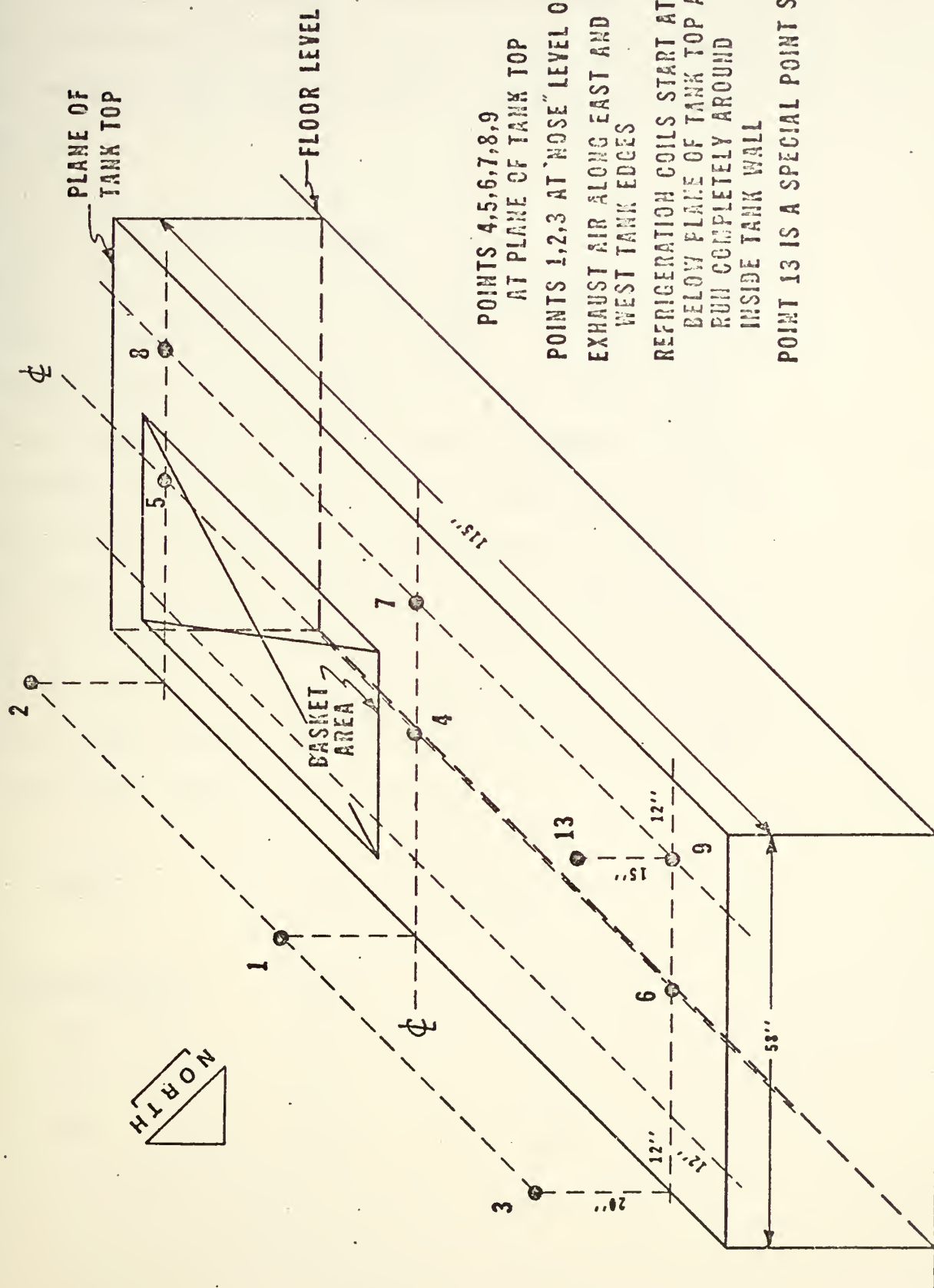
2. The sniffer was placed at the points shown in Figure 12. The three points along the east edge of the tank were placed at about the nose level of workers. The other points were located at tank-top level, with the exception of position 13 which is a point to which the sniffer was raised because of a high level of MEK concentration found at position nine. Exhaust air slots were located along east and west tank edges at tank-top level, but air flow appeared to be quite small. The basket was raised and lowered during monitoring periods.

3. All positions gave airborne concentrations within the OSHA limit except positions six and nine. Position nine was so bad that concentrations were for the most part off scale, so after recording at tank-top level for about one-half hour, the sniffer was raised 15 inches above tank-top level to position 13. The concentration immediately dropped below OSHA limit value and stayed



FIGURE 12

DEGREASE TANK P7-2 ELECTRO PLATING SHOP LOCATION OF SNIFFER POINTS



POINTS 4,5,6,7,8,9

AT PLANE OF TANK TOP

POINTS 1,2,3 AT "NOSE" LEVEL OF WORKERS

EXHAUST AIR ALONG EAST AND

WEST TANK EDGES

REFRIGERATION COILS START AT POINT 22"

BELOW PLANE OF TANK TOP AND

RUN COMPLETELY AROUND

INSIDE TANK WALL

POINT 13 IS A SPECIAL POINT SEE WRITEUP





there for the duration of the monitoring period. The signals were all quite noisy, probably due to complex air current movements over the tank. Raising and lowering of the basket was picked up by the sniffer, particularly if the basket was left in the raised position for any length of time. Typical results are shown in Figures 13, 14, and 15.

4. The next work planned in this area was to sniff for HCN gas over cyanide, cyanide rise, and cyanide sump tanks. The possibility of HCN exists for two main reasons. In the process of electroplating parts, they are dipped into acid, rinsed, and then dipped into cyanide solution, or vice versa, so pockets of acid may contact cyanide solution and generate HCN. Also, aqueous solutions of cyanides hydrolyze slightly at room temperature to form small amounts of HCN. The solution temperature, pH, and sodium hydroxide content largely determine the rate of hydrolysis. Hydrolysis rates increase appreciably with temperature. Also, there is a pronounced variation in HCN concentration above cyanide solutions with change in pH. This can be seen from the following:

NaOH	HCN Gas Above Solution *	
<u>In Solution (%)</u>	<u>mm Hg</u>	<u>PPM</u>
0.6	0.13	171
1.0	0.075	99
2.0	0.025	33







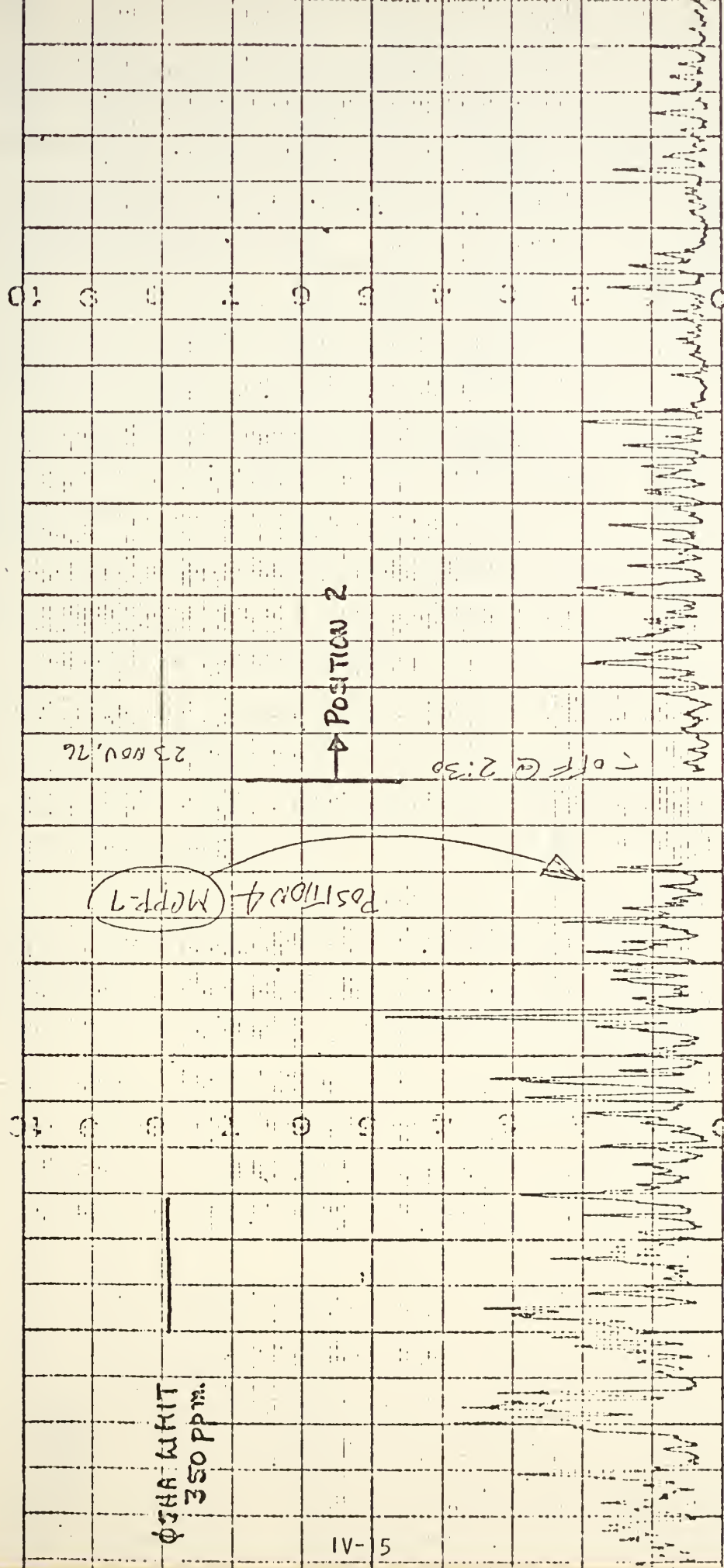


FIGURE 14

MIRAN I Output-Methyl Chloroform Concentrations At Positions 2 and 4 of Tank P7-2  
Electroplating Shop





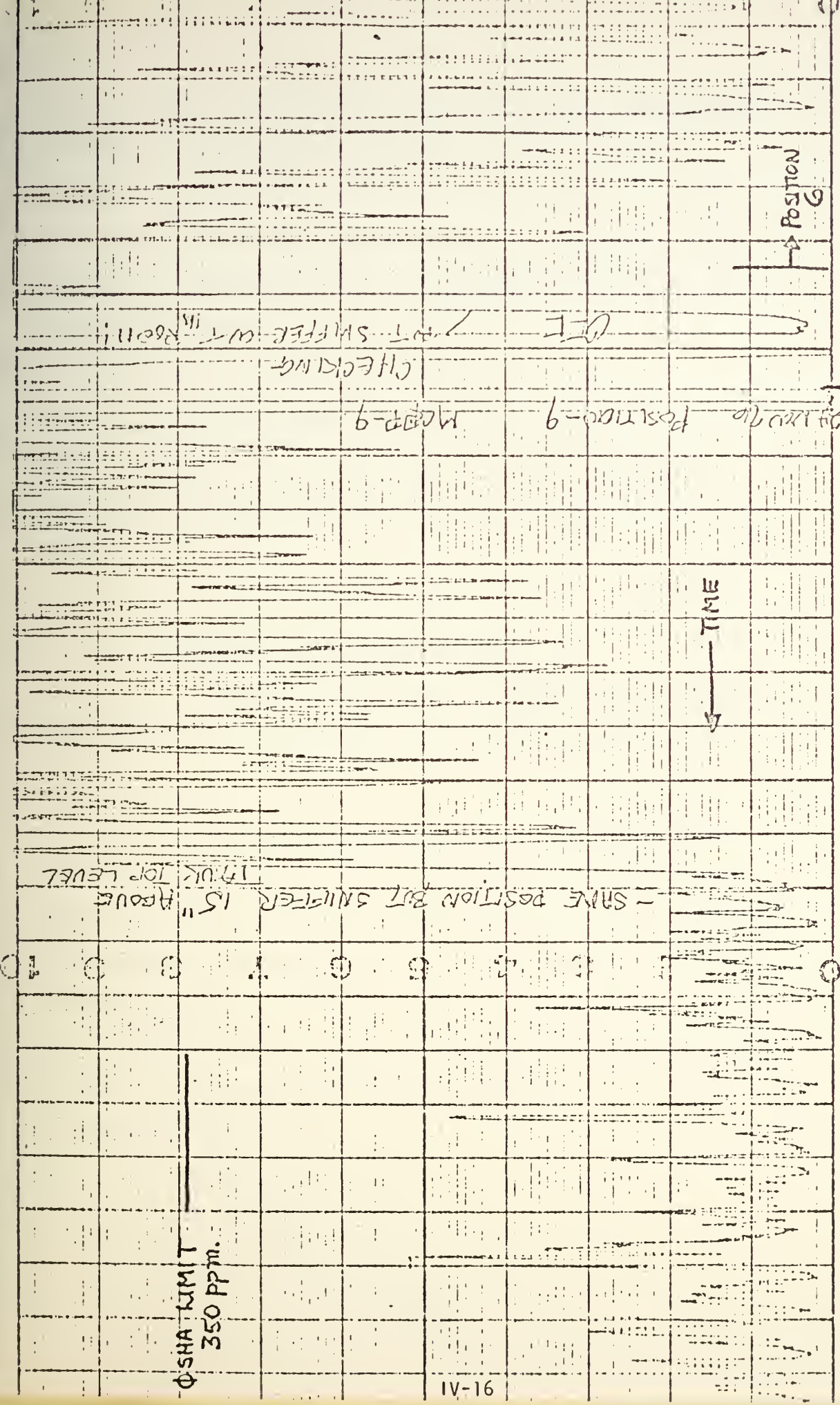


FIGURE 15

MIRAN | Output-Methyl Chloroform Concentrations At Positions 6 and 9 Of Tank P7-2  
Electroplating Shop





where the concentration of HCN is that which could, theoretically, accumulate in an enclosed space over solution at 122°F.

5. Obviously, good ventilation is needed over cyanide tanks and the solutions should be stabilized with about 2% sodium hydroxide (pH 10.4).

6. Unfortunately, because of inability to calibrate the MIRAN I for HCN, it was not possible to use the instrument for sniffing the electroplating cyanide tanks and sump areas for HCN vapors.

#### D. Discussion - Electroplating Shop

1. Since the only material successfully monitored was methyl chloroform, the discussion will concern this material.

2. The refrigeration coils do an acceptable job of trapping the rising vapors from the particular degreasing tank located in the Electroplating Shop. The vapor spillage in one cover of the tank was probably due too an abnormal condition such as a blockage of refrigerant in that area, or some other condition. The vapors were spilling at a point where workers do not normally spend any time. Any vapor spillage should be stopped once it is known to be occurring.



3. The general air movement in this shop is such that spilling vapors are promptly swept out of the building. There would be no tendency of the vapors to accumulate in the shop basement because of the air movement. However, there should be no spillage of vapor.

4. The value of the MIRAN I is that it is quite capable of detecting vapor "leaks" from tanks such as these.



## V. CONCLUSIONS AND RECOMMENDATIONS

Conclusions cover two areas. The use and performance of the MIRAN I Portable Infrared Gas Analyzer and the results obtained using this instrument.

The Analyzer is a dependable, accurate detector for four of the five materials investigated. It probably would work for HCN vapors at higher concentrations than those specified by OSHA limits. It is essentially a high level scientific instrument whose construction is rugged enough to allow its use on site in industrial areas. Its use for a variety of vapors requires some careful planning in that precalibration work off site must be done and then rechecked on site before use. Once the instrument is in place and monitoring, it requires very little attention and faithfully reports what it "sees". To change from one vapor to another is not a simple matter of changing some settings and away we go. A check calibration must be done for each vapor desired and this does take some time, about 30 minutes to one hour. Upon completion of the check calibration, the instrument is ready for monitoring.

Monitoring is quite good. The instrument time-constant is such that it picks up most high speed vapor concentration changes. It is also very specific for a given vapor, and generally screens out signals from other materials. This is not an absolute statement, however, since it is possible that another vapor or vapors could



give a signal at the settings for a specific vapor being monitored. If the presence of such other vapors were suspected, very careful work would be required.

The monitoring results in the Blade Bonding and Electroplating Shops indicate that, for the most part, vapor concentrations seen by the human "nose" are within OSHA limits. There are occasional peaks that go above these limits, but the long-time average is well within these limits. There is a good deal of dependence of vapor concentration on how people handle the various materials. The handling tends to be a bit careless, but that is a subjective conclusion. Almost surely, shop people and their supervisors will contend that their handling techniques are indeed safe and responsible. No matter how materials are handled, the MIRAN I can give a "picture" of what is going on in a given shop area, vapor wise that is.

It is recommended that the MIRAN I be used to study the presence of other vapors in the Blade Bonding and Electroplating Shops, and to monitor any vapor desired in other shops than these two. The only limitations on the instrument use are obtaining pure material for calibration purposes and knowing what materials are present if a complex chemical reaction is involved. For instance, the baking of bonds in the Blade Bonding Shop can possibly release various amines, isocyanates, etc., which could be harmful to humans who work right next to these areas. Study of the bonding reaction is recommended so as to indentify materials given off and then to arrange monitoring by use of the MIRAN I. A general review of all





areas where the instrument could monitor vapors is also recommended.

For analysis for HCN, it is recommended that wet chemistry methods be used until such time as some instrument with detectability limits on the low side (0-10 ppm) might be available. For quick and rough determinations of HCN vapor concentrations, it is recommended that the sensitive crayons manufactured by the Aromil Chemical Company, Division of Synorganics, Inc., P. O. Box 7627, Baltimore, Maryland 21207 be used; or the bit more sophisticated procedure of the hazardous gas detector, Model 8014, manufactured by Matheson Gas Products, a division of Will Ross, Inc., P. O. Box 85, East Rutherford, New Jersey 07073, be used. Copies of both of these procedures appear in Appendix D.



APPENDIX A  
MIRAN I LINEAR ABSORBANCE UNIT  
INSTRUCTION BOOK



WILKE SCIENTIFIC  
CORPORATION

140 WATER STREET • BOX 449 • SOUTH NORWALK • CONNECTICUT 06856

MIRAN-1 Linear Absorbance Unit  
Instruction Book

November 22, 1974

TELEPHONE (203) 853-1616 • TWX 7104683206



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## INTRODUCTION

The MIRAN-I Portable Gas Analyzer, shown in Figure 1-1, is a single-beam, variable filter, spectrophotometer, scanning the infrared spectral range of 2.5 to 14.5 microns, in conjunction with a 5.6 liter capacity cell whose pathlength is variable from 0.75 to 20.25 meters.

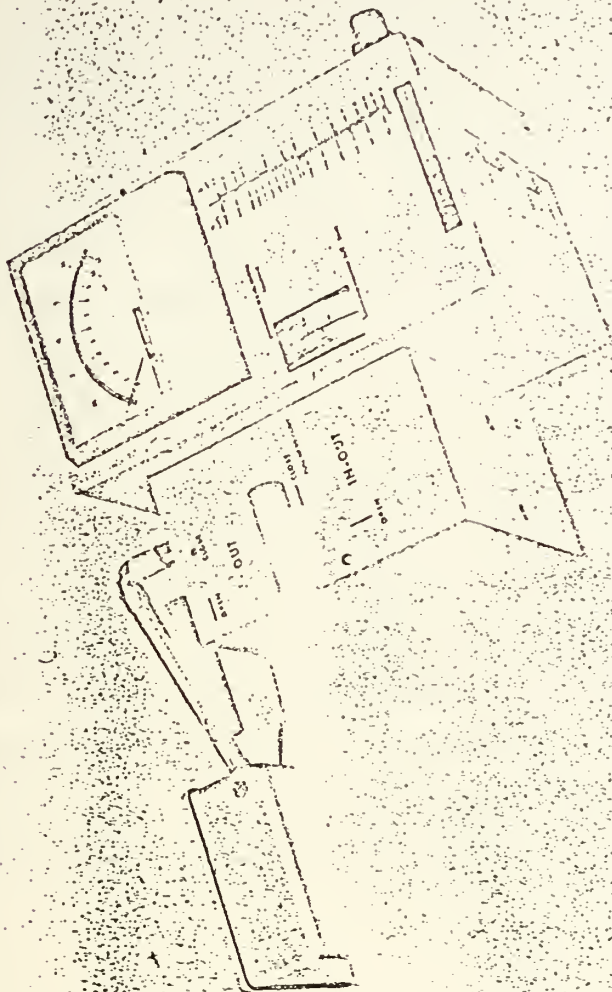
The MIRAN-I Portable Ambient Air Analyzer is equipped with a pump and ten foot sampling hose for drawing in air samples and a carrying case but is otherwise identical to the Gas Analyzer. Interchangeable filters for single wavelengths of interest may be used in another version of the Analyzer with the variable pathlength cell. The Analyzer may be converted for use with other cells for gas, liquid, or solid sampling when used with a (part no. 5600) base plate.

For increased versatility, a Strip Chart Recorder and Digital Display may be added to the basic analyzer. See price list for a listing of accessories, spare parts, and adaptor kits.

The Gas Analyzer System consists of two components, the cell and the analyzer, (or Infrared head), as itemized below.

The Variable Pathlength Cell consists of a 5.6 liter body, vacuum-tight to  $10^{-5}$  torr and pressurizable to 10 atmospheres; an internal optical path variable in 1.50 meter increments from 0.75 meters to 20.25 meters; a pair of windows transparent to infrared energy in the 2.5 to 14.5 micron band; inlet and outlet valves; and a safety valve. The internal optics are gold plated and the inside of the cell is Teflon coated to resist sample absorption and corrosion.





MIRAN I GENERAL PURPOSE GAS ANALYZER  
WITH AMBIENT AIR PUMP

FIG. 1-1



The Analyzer consists of a radiation source; mirror system; mechanical chopper; circular filter, variable in three segments from 2.5 to 14.5 microns; a scanning motor, for automatic spectrum analysis; pyro-electric detector; a signal preamplifier; logarithmic range-compensating circuitry; regulated power supplies; and a meter providing absorbance and percent-transmission scales, and a 0-1 volt output for use with a strip chart recorder or Wilks Digital Display.

The MIRAN I system operates from either a 115 V or 230 V, 50-60 Hz power supply. By means of an inverter (Part No. 5631), portable operation from a 12 volt battery, vehicular type, is readily accomplished. Part No. 5629 is an inverter system complete with battery and charger. This expands the instrument's use to monitoring beyond the confines of the laboratory and greatly facilitates the determination of environmental pollutants and in meeting OSHA (Occupational Safety Health Act) industrial requirements.





## SPECIFICATIONS

1-2

### SPECTROMETER (TYPE 5633)

Type	Single-beam spectrometer
Wavelength Range	2.5 to 14.5 microns in three steps of 2.5 to 4.5, 4.5 to 8, and 8 to 14.5 microns, with small overlaps.
Wavelength Range Control	Manual, or motor-driven scan of 9 minutes.
Resolution (Approximate)	.05u at 3 microns, .12u at 6 microns, .25u at 11 microns.
Noise Level	$1 \times 10^{-4}$ Absorbance Units, maximum, under following conditions: Without cell, 1 mm slit, 1 second TC, 3.5 microns wavelength, 23°C Temperature.
Drift	.004 Absorbance Units, maximum, at 3.5 microns wavelength, 23°C temperature.
Photometric Accuracy	Better than .5%
Slit Width Range	.25, .5, 1.0, 2.0 millimeters, and closed.
Time Constant Range	.25, 1.0, 2.5, and 10 seconds.
Absorbance Ranges	0-.025, 0-.1, 0-.25, 0-1, Absorbance Units, full-scale, and 0-100% Transmission.
Wavelength Drive Speed	2.5 minutes per segment.
Infrared Source	Regulated Nichrome wire heating element.
Infrared Detector	Pyroelectric type. Lithium Tantalate element.
Power Requirements	25 watts at either 115 or 230 VAC at 50-60 Hz.
Weight	12.5 pounds (5.8 Kg) without cell. 30.0 pounds (11.6 Kg) with 20 meter cell.
Dimensions	without cell (24.5 x 15.5 x 15.5 cm) with 20 meter cell (70 x 28 x 18 cm)
Temperature Range	32° to 104°F (0° to +40°C) Operating -4° to 140°F (-20° to +60°C) Storage



SPECIFICATIONS (Continued)

VARIABLE PATH 20-METER GAS CELL

Pathlength	.75 meters to 20 meters or more in steps of 1.5 meters, externally set.
Volume	5.6 liters
Pressure Range, Operating	$10^{-5}$ torr vacuum to 10 atmospheres.
Pressure Range, Maximum	Safety valve set for 150 lbs/square inch. (10.5 Kg/cm <sup>2</sup> )
Valves	Inlet and exhaust valves designed for both vacuum and pressure, Teflon sealed.
Internal Finish	Teflon lined, with mirrors and other components gold plated.
Windows	NaCl normally furnished. Windows of AgBr available.



## ANALYZER

The Analyzer controls are shown in Figures 1-2, 1-3, and 1-4.

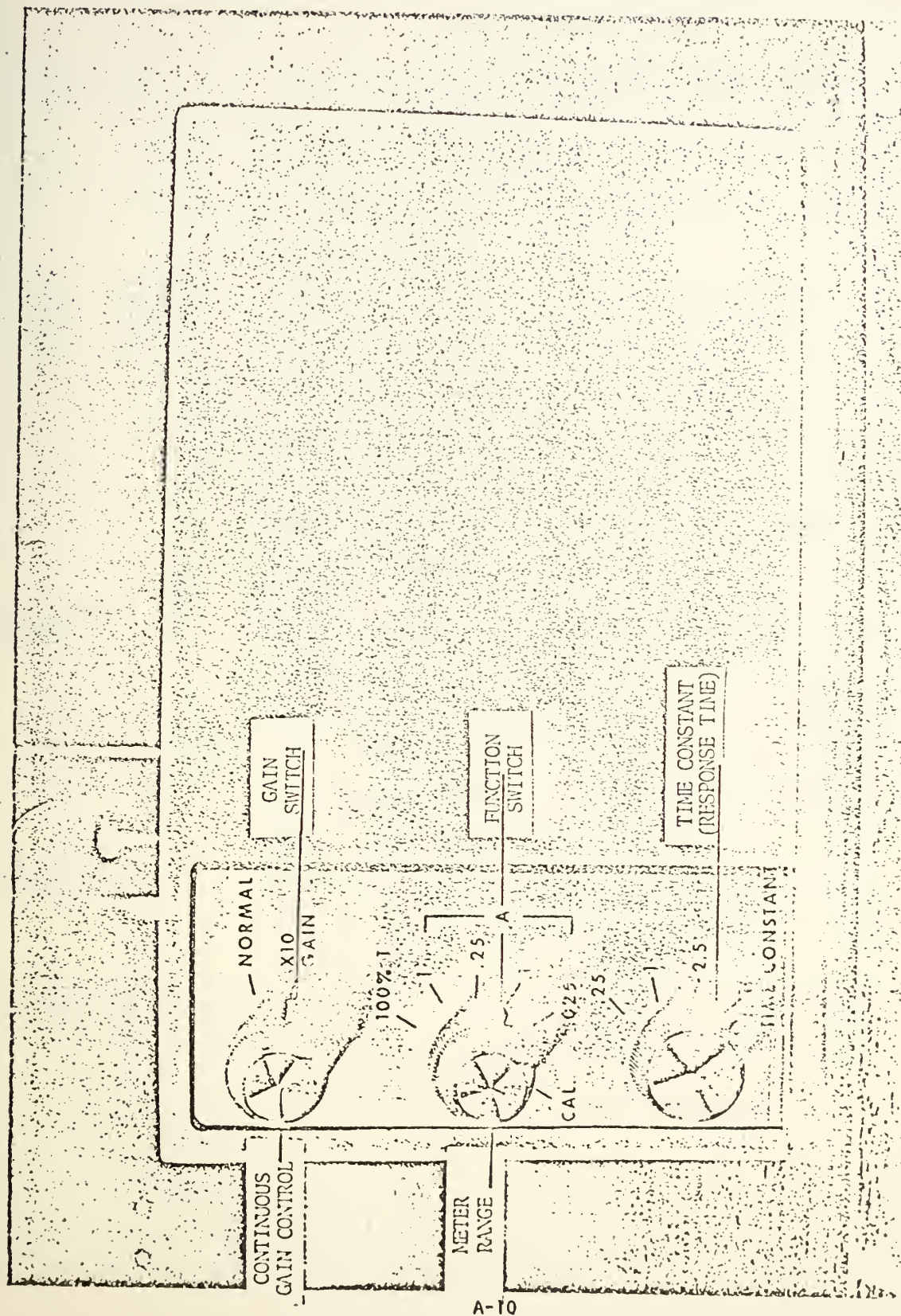
CONTROL TITLE	FUNCTION
POWER	Power switch for entire Analyzer.
Pilot Light	When POWER switch is on, pilot light illuminates red.
SCAN	Switch for scanning motor. Entire range may be scanned in nine minutes. May be over-ridden for hand scanning at any time through slip-clutch.
SLIT	The Slit Width Selector governs the radiation output of the infrared source in five steps: Closed, .25mm, .50 mm, 1.0 mm, and 2.0 mm. For normal operation, the 1 mm slit is generally used.
WAVELENGTH	The Wavelength or Filter Wheel determines the radiation wavelength being measured, in three ranges of 2.5 to 4.5 microns, 4.5 to 8.0 microns, and 8.0 to 14.5 microns. The red portion of the control indicates that unfiltered energy is being passed. This is useful for purposes of optical alignment, but should not be left continuously in this position, to avoid detector overload.
Gain Controls	These two controls are co-axially mounted.
Gain	The central knob is the GAIN Control, which varies the detector signal amplification. It is used for adjusting the Absorbance and transmission ranges on the meter.











MIRAN I ANALYZER CONTROLS

FIG. 1-3



Gain Switch	The Gain Switch in the NORMAL position affects the Gain Control by a factor of 1. In the 10X position, it amplifies the Gain Control setting by a factor of 10. The 10X position is required for many applications.
Range Controls	These two controls are co-axially mounted
Meter Range	The central control (screw-driver slot on some models) allows the full-scale reading of the meter to be set to values other than the calibrated fixed ranges. For most applications the control should be left in the <u>Calibrated</u> position (fully counterclockwise).
Absorbance 1% T Range	The outer knob selects the mode of Analyzer operation. The % T position causes the meter to read linearly in percentage of transmission.
(Function Switch)	Four Absorbance ranges are provided for setting 0-1, 0-0.25, 0-0.1 and 0-0.025. The meter will read linearly in Absorbance units.
RESPONSE TIME	This switch provides four time constants of .25, 1.0, 2.5, and 10.0 seconds, yielding an integrated signal over the interval selected.
Recorder Connections	The red (+) and black (-) binding posts provide an output of 0 to 1 volt range, suitable for a chart recorder or analog-to-digital meter.



#### Meter

The meter provides dual scales. The lower scale which reads from 0-1.1 is read when using the % T, 0-1, or 0-0.1 absorbance ranges. The upper scale which reads from 0-0.26 is used for the 0-0.25 and 0-0.025 Absorbance ranges.

#### Fuse

Type AGC. Use 1 Amp fuse for 115 VAC and 1 Atp for 230 VAC operation.

#### Conversion Chart

A chart, giving wavelengths from 2.5 to 15 microns and corresponding wavenumbers from  $4000\text{ cm}^{-1}$  to  $660\text{ cm}^{-1}$ , is located on the top of the Analyzer below the meter.



## GAS CELL

The Gas Cell controls are shown in Figures 1-4 and 1-5.

### CONTROL TITLE.

### FUNCTION

#### PATHLENGTH

This 14 turn dial varies the pathlength of the radiation beam from 0.75 meters to 20.25 meters, in 1.5 meter increments. See Table I.

#### PRESSURE RELIEF VALVE

To protect the cell from pressures exceeding ten atmospheres, the safety valve will "blow out". It remains in the open position until reset.

#### Sample Valves

There are two valves, one marked IN-OUT, which may be used for both admitting and removing samples from the cell, and an OUT valve, for sample removal only. The bottom tube is the IN-OUT valve port, the top tube for the OUT valve. To prevent damage to the Teflon valve seals, the valves should be left in the fully closed or opened positions only.

#### Carrying Handle

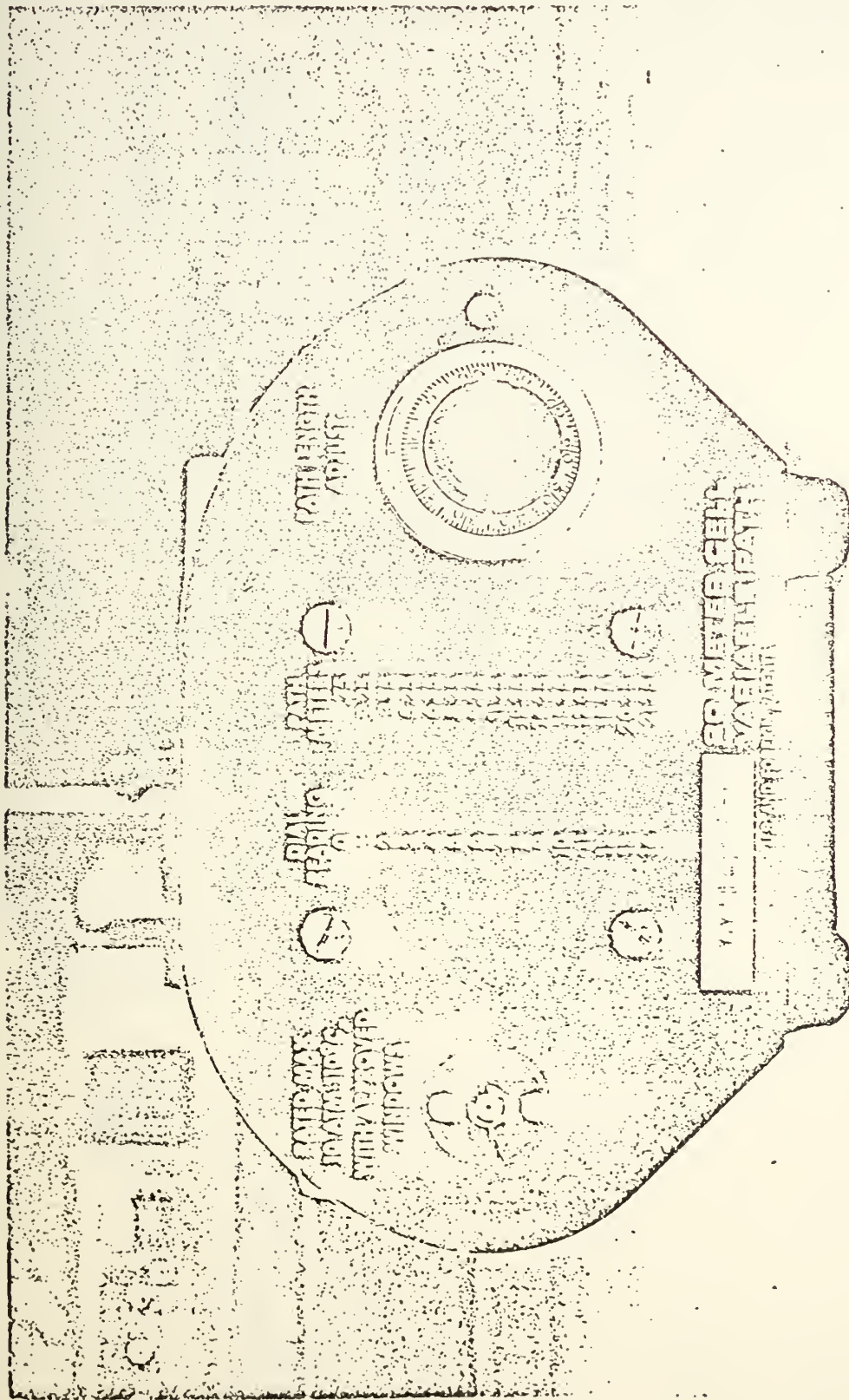
To carry the Analyzer, close the IN-OUT valve, then pull up on the handle about one-half inch, where a detent will lock it into place.

#### Pump

The Ambient Air Analyzer is equipped with a self-contained electric pump, operating from the same supply as the instrument. The power switch is located on cover housing the pump.



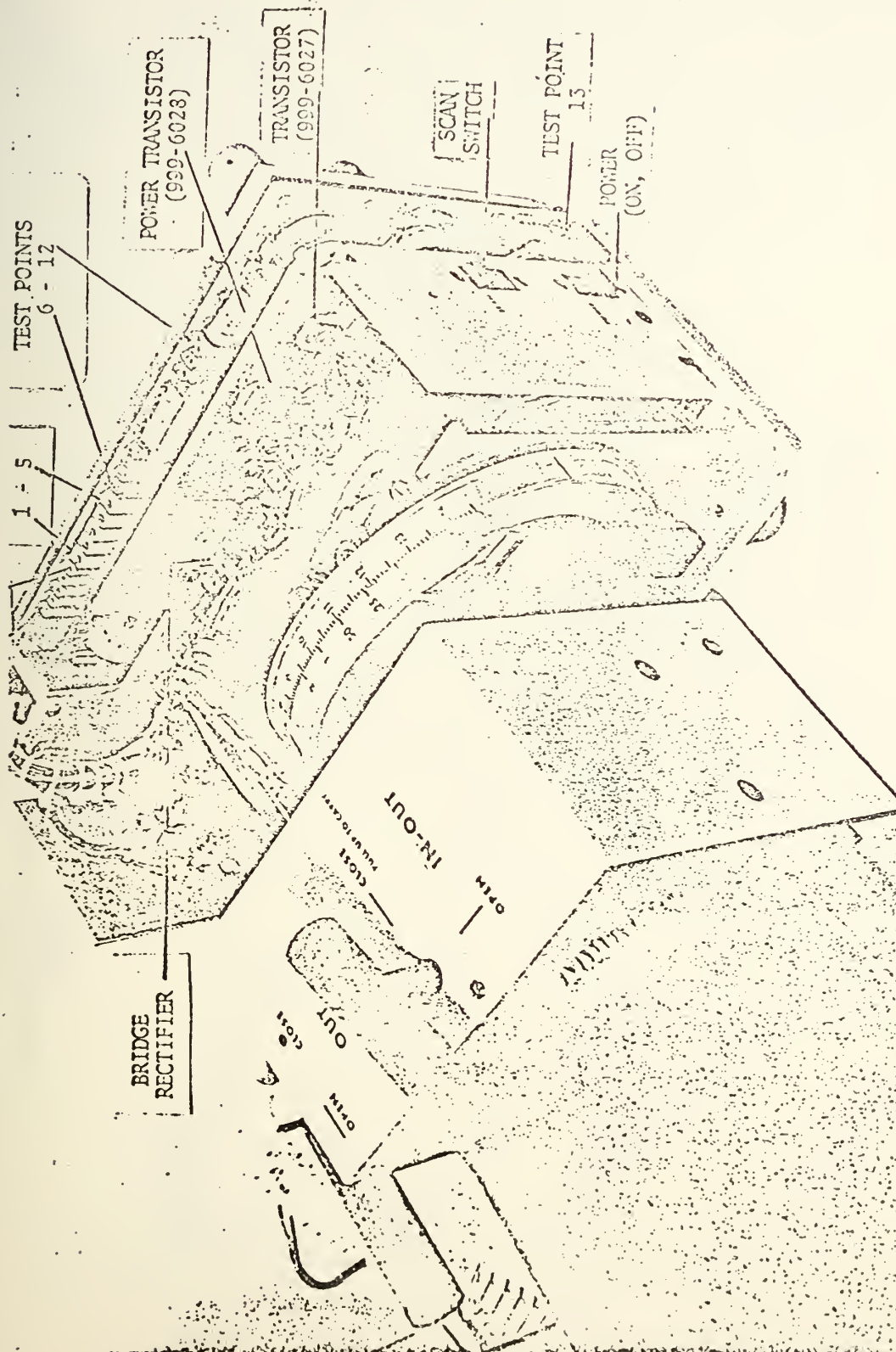




MIRAN I GAS ANALYZER (END VIEW)

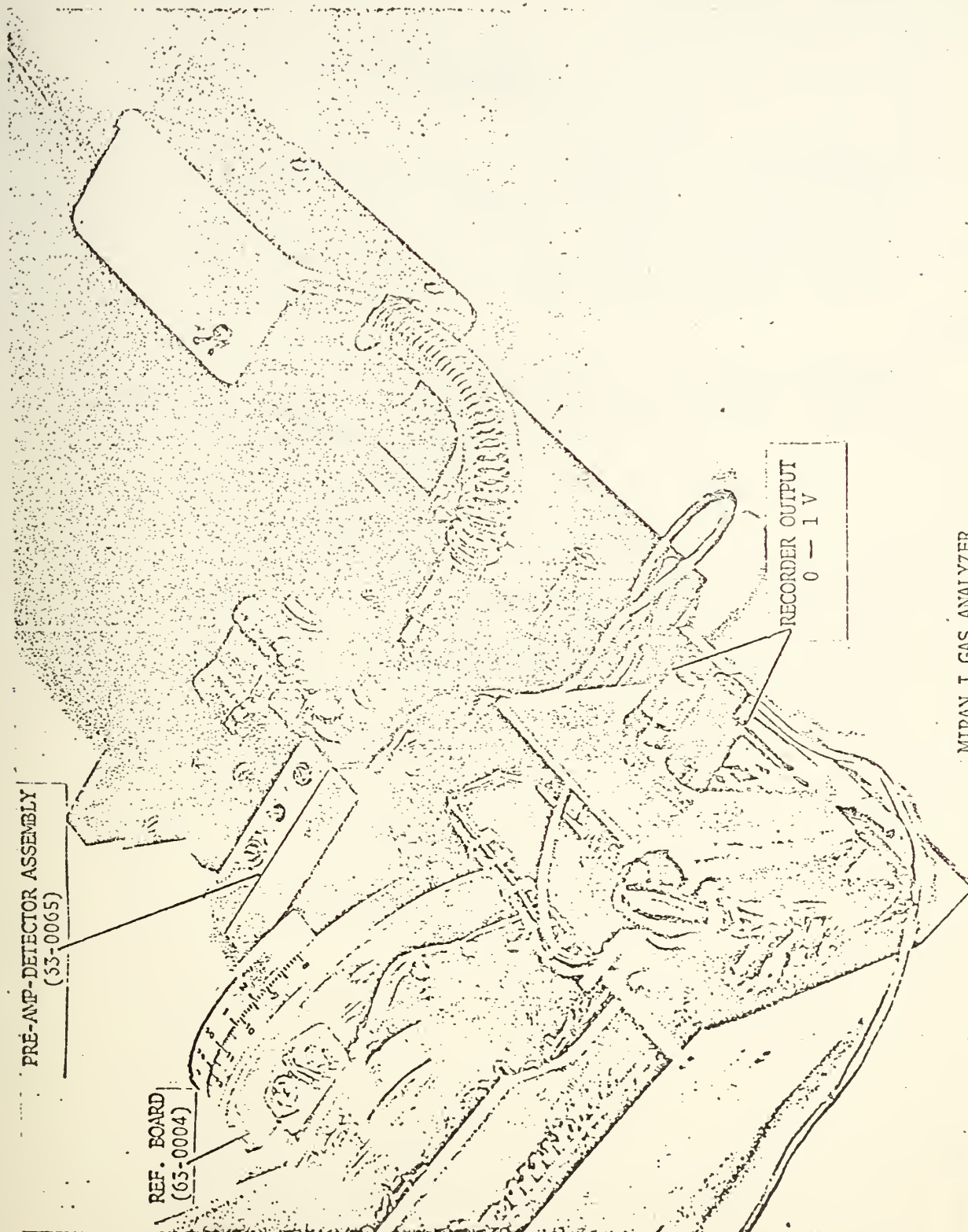
FIG. 1-4





MIRAN I GAS ANALYZER SHOWING INTERNAL COMPONENTS OF THE SPECTROMETER AND VALVES ON THE GAS CELL





MIRAN I GAS ANALYZER  
WITH COVERS REMOVED

FIG. 1-6





## SECTION II.

### INSTALLATION

#### 1 UNPACKING

Instruments are carefully packed at the factory. If a carrying case (No. 5714) was ordered with the analyzer, (included with the Ambient Air Analyzer) the apparatus will be inside it for additional protection. The MIRAN I Gas Analyzer will include the following items, subject to modifications:

AMOUNT	ITEM DESCRIPTION	PART NO.
1	MIRAN I Portable Gas Analyzer	5652 (Old No 5633)
1	Instruction Manual	
1	AC Supply Cord	

WE HAVE THIS

The MIRAN I Air Analyzer will normally include the following items:

1	MIRAN I Portable Air Analyzer	5653	
1	Carrying Case	5714	} Included in 5653 package.
1	Air Pump	5711	
1	Air Hose	5712	
1	Instruction Manual		
1	AC Supply Cord		





After determining that all parts listed above are present, make an inspection for visual damage. If any damage is present, notify the Carrier and request an inspection report. Save all shipping containers and documents for the inspection, and be sure that the Carrier furnished a signed copy of his report.

#### Location

If the analyzer is to be used in conjunction with other apparatus in a generally fixed location, ensure that it will be free from dampness, drafts, temperature extremes, vibrations and high noise levels, and from power line surges and fluctuations exceeding its operating limits. Normal operation of the analyzer should not cause electro-magnetic radiation or interference to other apparatus.



## INITIAL CHECKOUT

1. Before applying power, ensure that the voltage selector is in the correct position, either 115 VAC or 230 VAC, for the line voltage supply. The switch is located above the power cord receptacle. The supply outlet should be of the three-wire grounded type.
2. Throw the POWER switch to ON, and allow the instrument to warm up for five minutes.
3. Set the RANGE selector to %T, and the SLIT selector to CLOSED. The meter should read approximate zero. If it does not, check with Wilks Scientific.
4. Open the slit to the 0.5 mm setting.
5. Set the WAVELENGTH dial to 3.5 microns.
6. Set the Gain Switch to X 10.
7. Set the time constant selector to .25 .
8. Turn the PATHLENGTH dial to 0.000 .
9. Adjust the continuous GAIN control (central knob) for a meter reading of about 60% transmission. (Change gain switch to normal if needed)
10. Rotate the PATHLENGTH dial counter-clockwise one turn. The dial should now read 14.00, and the meter should indicate considerably lower than 60%.
11. Slowly rotate the PATHLENGTH dial clockwise. The meter reading should now increase, going through a maximum when the dial is near zero (between 14.50 and 0.50) and then decrease. Note this maximum meter reading. Rotate the dial counter-clockwise back to 14.00, then clockwise, until the maximum reading previously noted is again obtained.



12. Turn the continuous GAIN control counter-clockwise to its minimum setting. There is no fixed stop as such on this control, but the knob will become harder to turn, and the meter will no longer vary, once the minimum setting has been reached.
13. Check that the meter reading is now within 5% of that for Test A on the enclosed Instrument Test Sheet, furnished with all new instruments. For equipment that has been in service for some time, values as low as one-fourth of the Test Sheet values are still acceptable.
14. Maintaining the settings of Step 12, turn the GAIN control clockwise until the meter reads 100 %T.
15. Turn the PATHLENGTH dial clockwise to about a setting of 13. The meter will go through a maximum with each revolution of the knob. The transmission maximum reading that corresponds to a pathlength of 20.25 meters should occur near that dial reading given for Test B on the Test Sheet. Set the pathlength to this maximum, while rotating the knob clockwise, as described in Step 11. Due to mechanical backlash in the system, it is desirable to continue to turn the dial a full turn, then reverse, to reach a point that has been just overpassed.
16. The meter reading should agree within  $\pm 5\%$  of Test B of the Test Sheet. Equipment in use may show a value as low as one-fourth of the Test B reading.
17. Open the slit to 2mm, and set the WAVELENGTH dial for 12 microns. Repeat Steps 6 through 12 above. The meter reading should be within  $\pm 5\%$  of Test C of the Test Sheet. Instruments in service may show a value as low as one-half of the Test Sheet reading.



18. Repeat Steps 14 and 15. The meter reading should be within  $\pm 5\%$  of Test D of the Test Sheet. Instruments in service may show a value as low as one-half of the Test Sheet reading and still be acceptable.

TABLE 2-1

Nominal Readings of Pathlength Dial (n) VS Cell Pathlength (L)

<u>Pathlength Dial*</u>	<u>Cell Pathlength (Meters)</u>	<u>Pathlength Dial</u>	<u>Cell Pathlength (Meters)</u>
0.00	0.75	8.00	12.75
1.00	2.25	9.00	14.25
2.00	3.75	10.00	15.75
3.00	5.25	11.00	17.25
4.00	6.75	12.00	18.75
5.00	8.25	13.00	20.25
6.00	9.75	14.00	21.75
7.00	11.25		

\* Allow  $\pm 0.30$  on the dial reading.

Figure provides a graph of meter readings in % T versus PATHLENGTH dial reading, showing nominal and actual dial readings for a particular cell. Actual dial readings for different will normally be within  $\pm 0.30$  of nominal values.





## SECTION III

### OPERATION

#### 3-1 GENERAL

It is important to know what type of environment the analyzer will operate in. A relative humidity exceeding 80%, for instance, will cause the gradual deterioration of the standard NaCl windows in the sampling cell. Likewise, if samples containing high water vapor content are to be regularly run, replacement of the salt windows with those made of silver bromide (AgBr) is recommended. Avoid, if possible, the placement of the instrument in drafts, or where nearby machinery may cause vibrations or a high noise level. Sampling of explosive atmospheres can be accomplished by employing an ambient air pump (such as is already installed on the MIRAN I Air Analyzer) and up to 200 feet of sampling hose to isolate the analyzer. The cell may also be replaced with other sampling systems. Consult Wilks Scientific for specific problems.

#### 3-2 PRELIMINARY STEPS

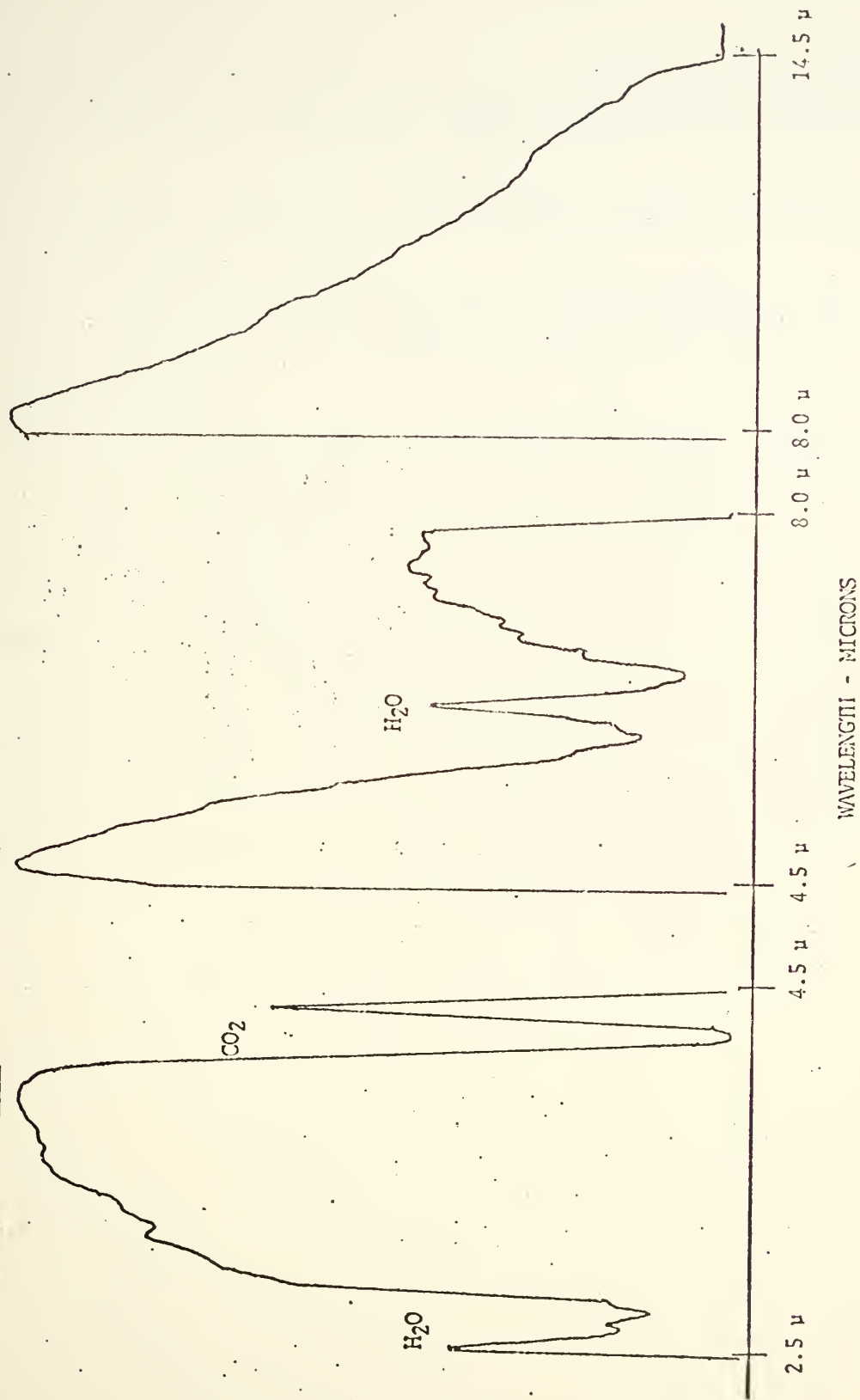
It is assumed that the operator has already performed the Initial Checkout steps of Section 2-3. The following procedure may be done at the start of each day's operations.

1. Check for correct setting of the Voltage Selector switch.
2. If a 12 volt storage battery is to power the analyzer for



# MIKAN SPECTRUM OF AMBIENT AIR

100% T



NOISSINSVIL :  
A-23

FIG. 3-1



portable operation, make sure of its charge condition and that the Inverter (No. 5631 or No. 5629) is delivering its rated output.

3. With the SCAN switch off, turn the POWER switch on and allow a fifteen minute warm-up.
4. Set the RANGE selector to  $\frac{1}{2}$  T and the PATHLENGTH dial for 20.25 meters. The SLIT should be set at 1 mm, and the Gain switch at NORMAL.
5. Vary the WAVELENGTH dial from 2.5 to 8.5 microns, observing that the point of maximum transmission on the meter falls to about 90%. If the maximum is greater or less than this, adjust with the GAIN control.
6. Return the WAVELENGTH dial to 2.5 microns and start the automatic scan. If a chart recorder is available, the spectrum of ambient air should appear as in Figure 3-1. Published double-beam spectra, referenced in the Bibliography Section, are still applicable for this single-beam instrument, and are useful in checking for interferences from ambient contaminants.



## CELL CALIBRATION FOR QUANTITATIVE ANALYSIS

### GENERAL

For accurate quantitative analysis, it is necessary to calibrate the analyzer for each sample wavelength. However, this is a one-time procedure, unless the wavelength filter is replaced or a new cell is installed.

Calibration may be accomplished by several methods. The following technique has been developed at Wilks Scientific and is shown schematically in Figure 3-2. It is available in kit form as Wilks Part No. 5707, consisting of a stainless steel bellows pump, septum stainless steel fittings and Teflon connecting tubes. In operation, calibrated samples are introduced through a septum by gas or liquid syringes and circulated through the cell by means of the pump.

Liquid samples are vaporized in the process of circulation, their concentrations, in parts per million, being determined by the following formula, where Ideal Gas Laws in the total volume of the system may be assumed.

$$C \text{ (ppm)} = \frac{\rho V}{M} \times \frac{(RT)}{(P)} \times \frac{10^3}{5.64} \quad \text{where:}$$

$V$  = Sample volume in micro-liters (Cell volume = 5.64 liters)

$\rho$  = Liquid density (gm/cm<sup>3</sup>)

$M$  = Molecular Weight of sample

$\frac{(RT)}{(P)}$  = Molar volume of gas (24.4 at 25° C) & 1 atmosphere

-----  
For gases,  $C \text{ (ppm)} = \frac{v}{5.64}$  where  $v$  = sample volume in micro liters.





## 1 CALIBRATION PROCEDURE

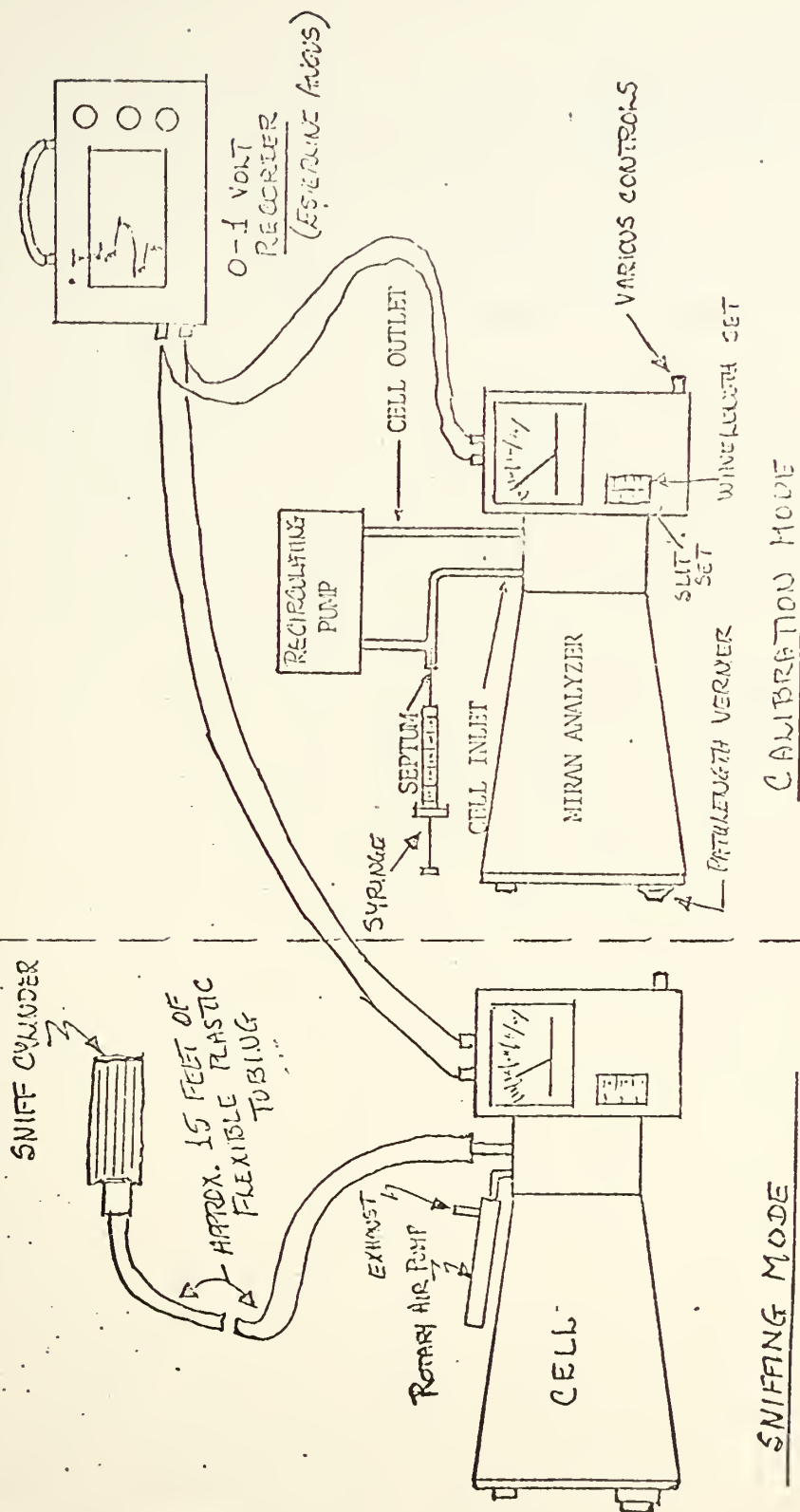
1. It is important that the cell be free from all contaminants. Flush the cell with clean air, evacuate, and flush again. Perform a scan as described under Initial Checkout in Section 2-3.
2. Connect the outlet of the pump to the cell's IN valve, and the pump inlet to the OUT valve, as shown in Figure 3-2. A recorder having a 0-1 volt range may be connected to the analyzer's red (+) and black (-) binding posts. Turn on bellows pump.
3. If the analytical band and strength of the sample is unknown, the OSHA wall-chart may be used to find the absorbance wavelengths and pathlengths for analysis near the TLV\* limits. Use a slit width of 1 mm, a pathlength of 20.25 meters, and Absorbance range, best suited for reading the Absorbance value
4. Inject a sample by syringe into the septum. Typically, this will be about 5 micro-liters of liquid or 500 micro-liters of gas. Liquid samples should be injected slowly to ensure complete vaporization.
5. After 30 seconds to one minute, depending upon when the absorb-reading has stabilized, turn off the pump and scan the spectrum from 2.5 to 14.5 microns. Locate the strongest absorption wavelength, carefully rocking the dial to determine the maximum absorbance. Note, that if the cell contains ambient air, there will be CO<sub>2</sub> and H<sub>2</sub>O bands also present, as shown in Figure 3-1. If these bands can be expected to interfere with locating sample bands then the cell may first be flushed with dry nitrogen.

\* TLV = Threshold Limit Value, from the OSHA health-safety requirements.



FIGURE 3-2

SCHEMATIC DIAGRAM OF CLOSED LOOP CALIBRATION SYSTEM & SNIFFING MODE





6. Flush the sample out of the closed loc. system and gas cell with clean air or "zero gas" such as that obtainable when using the Wilks "zerogas" filter (part No. 5660).
7. Close the slit (0) and set the analyzer function for % T. Check the zero adjustment on the recorder to insure that both the meter and recorder read zero.
8. Reopen the slit (usually 1mm) and adjust the continuous instrument gain to read 100 % T (the gain switch will usually need to be in the X10 position.) Adjust the recorder if necessary to read 100 units as well. This adjustment is the attenuator adjustment on (Wilks part No. 3990). LDC Model 320 recorder.
9. Change the analyzer function to the Absorbance range best suited for the calibration. The meter will read at or near zero. Re-adjust continuous gain if necessary to set zero precisely. Select a response time (time constant) that give an acceptable noise level (2.5 sec. or 10 sec. are most frequently used).
10. Turn on the bellows pump, and commence injecting equal sample increments (one micro-liter for liquids and 5 or 50 microliters for gases being typical) pausing about 30 seconds between injections. A 5.64  $\mu$ l injection of a pure gas will result in a 1ppm mixture of gas in the cell and calibration system because the volume is 5.64 liters (5.6 liters for the cell, 40 ml for the calibration system).
11. Flush the sample out with clean air or "zerogas". For Ambient air studies, room air is often employed as a zero gas, but care must be taken through good ventilation not to build up a sample "background" in the vicinity. Use of the "zero gas filter" is adviseable.



12. By means of a recorder chart (example figure 3-3A) plot an Absorbance - Concentration calibration curve. Figure 3-3B represents a typical calibration curve for vinyl chloride ( $\text{CH}_2\text{-CHCl}$ ) at a concentration of 0-5 ppm.

\*\* Note - Please refer to section 1 illustration 1-2, 1-3, and 1-4. These illustrations show typical instrument settings for calibration and in plant use of the analyzer for vinyl chloride measurement. Figure 1-4 shows the pathlength set at 20.25 meters for a typical instrument. Figure 1-2 shows the wavelength and slit set respectively at  $10.9\mu$  and 2mm. The meter is reading an absorbance of 0.05 Absorbance units. Figure 1-3 shows the function selector set at 0.1 Absorbance units full scale and the time constant set at 10 seconds. The X10 gain is the required position of the gain range switch.





## 1 ATMOSPHERIC SAMPLING

Atmospheric sampling requires only that samples be flushed into the cell for scanning. In as much as a large proportion of the analyzer's operations will concern a single material or contaminant, it will usually be set on the wavelength of the substance in question for observations of changes in concentration. Periodic flushings of the cell with zero (clean) air may be necessary to recheck the Absorbance zero of the Analyzer. Where there is local contamination, bottled air or a zero air filter such as part No. 5660 may be necessary for cell flushing purposes. Such situations may arise during analysis within closed environments like solvent tanks or storage areas or when checking for leaks in pipes or ductwork.

## 2 SELECTED WAVELENGTH ABSORBANCE MONITORING

Where a single wavelength is of interest, such as the presence or concentration of carbon monoxide in a tunnel, or measurement of vinyl chloride the following procedure may be employed.

1. The instrument should have first passed the Initial Checkout of Section 2-3.
2. Set up instrument for gas to be monitored (see wall chart)
3. Select a RESPONSE TIME setting that will give a smooth meter response without being unduely sluggish. See that the power supply is free from voltage surges and "spikes", which might cause erratic meter indications.



4. With clean air or "Zero gas" in the cell, adjust for a 100% transmission reading with the GAIN control. The Gain Switch should be at X10 for most applications.
5. With the Function Selector on the 0-1 Absorbance scale, the meter should read zero. To adjust for zero on all ranges, switch to the .025 setting and carefully vary the GAIN control.
6. Select the desired absorbance range for monitoring.
7. Open valves and switch on the ambient air pump continuously with the sample hose (Be sure to use the hose with a particulate filter).



## MEETING OSHA LIMITS

The Occupational Safety and Health Act of 1970 has set minimum tolerable limits of gas and vapor concentrations in working areas for some 400 materials. The MIRAN I air and gas analyzers are highly useful for on-site sampling to quickly and accurately determine whether the area tested meets OSHA limits.\*

Nearly all chemical compounds have characteristic infrared spectra which may be used for identification and to determine the amount present. While the infrared spectra of most compounds are complex each spectrum usually contains a small number (from one to five) of strong, identifiable bands. When spectra are measured at the low concentrations usually encountered in OSHA compliance testing, only the stronger bands appear, and the probability of more than one compound absorbing on the same wavelength is minimal. Even when there is interference between compounds, the absorbances are additive and the error is always in the direction of a greater concentration apparently being present. In no case is the observed concentration less than the actual value; consequently, where an absorbance less than the OSHA limit is observed, the actual value is also less than the limit.

A Wall Chart of gases and vapors for which toxic limits have been established, and including additional infrared analytical information, is to be found in the back cover of this manual. For each compound, the chart lists the maximum allowable exposure limit, in parts per million and in milligrams per cubic meter. Those compounds with limits within the sensitivity range of the MIRAN I analyzer are accompanied by the following additional information: analytical

\* See also Wilks Scientific Bulletin MI-4.



wavelength, minimum detectable concentration, absorbance reading at the OSHA limit, and the pathlength setting. The scale expansion factor need not be considered for current MIRAN I models, as this function is automatically performed by the "Log Board", described in Section 4-2-1.

Using the Wall Chart data, the wavelength and pathlength for the compound of interest are set on the analyzer, which is then zeroed with clean air. The sample is then admitted into the cell, according to the methods described in Sections 3-4-1, 5-3, 4-2. If the absorbance indicated is less than the OSHA limit, the sample location is "safe" as far as that particular compound is concerned.

The measurement technique is based on a "single point" straight line calibration plot, as shown in Figure 3-4. Although the actual calibration plot for most vapors is curved, for absorbances less than the OSHA limit, true concentrations may be less, but never greater than the "single point" value. For absorbances greater than the OSHA value, actual concentrations may be greater, but never less than the "single Point" value. Thus, the "single point" method may be used with complete confidence.

Some typical selections from the OSHA Wall Chart are shown below.





TABLE 3-1

Compound	Maximum Allowable Exposure (8 Hour Weighted Average)		PPM	mg/m <sup>3</sup>	Analytical Wavelength	Minimum Concentration Which the MIRAN Analyzer can Detect	Absorbance Equivalent to the OSHA Limit	
							Absorbance	Pathlength
Sulfur Dioxide	5	13			8.6/ $\mu$	0.14 PPM	0.02	20.25
Carbon Dioxide	5000	9000			4.25	0.05	0.12	0.075
Carbon Monoxide	50	55			4.7	0.2	0.04	20.25
Carbon Dioxide	50	35			10.4	0.9	0.07	20.25
Ethyl Alcohol	1000	1900			9.5	0.2	0.5	2.25
Acetone	1000	2400			8.2	0.09	0.49	2.25
Gasoline	100	360			8.9	0.05	0.38	5.25
Carbon tetrachloride	10	-			12.6	0.06	0.3	20.25
Carbon disulfide	1000	5600			11.9	0.01	1.7	0.75
Ethylene chloride	500	-			13.3	0.2	0.31	0.75
Hexachlorobenzene	300	1050			3.4	0.03	0.32	2.25
Chlorobenzene	10	-			3.3	0.15	0.017	20.25
Chloroethylene	100	-				0.2	0.29	5.25



FIG. 3-3A

Instrument  
response to successive  
concentration increases of 1  
ppm. Conditions: slit, 2 mm;  
pathlength, 20.25 m; time  
constant, 2.5 sec;  
wavelength, 10.9  $\mu$ m.

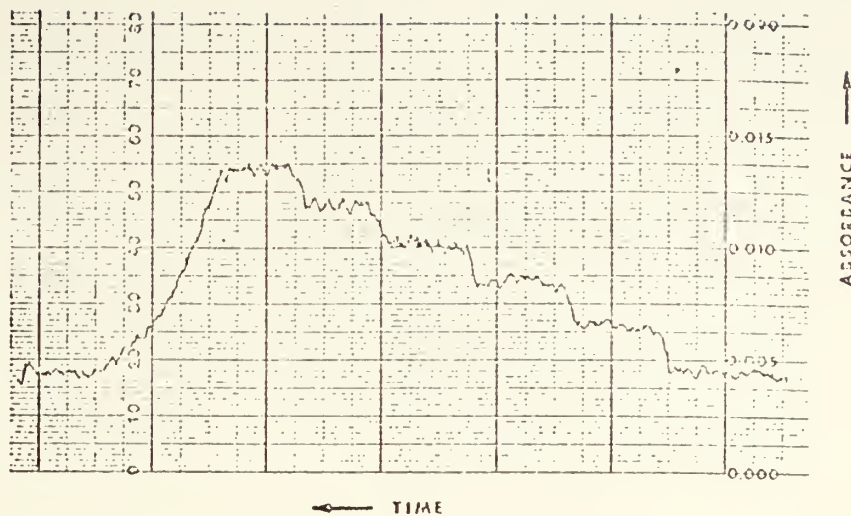
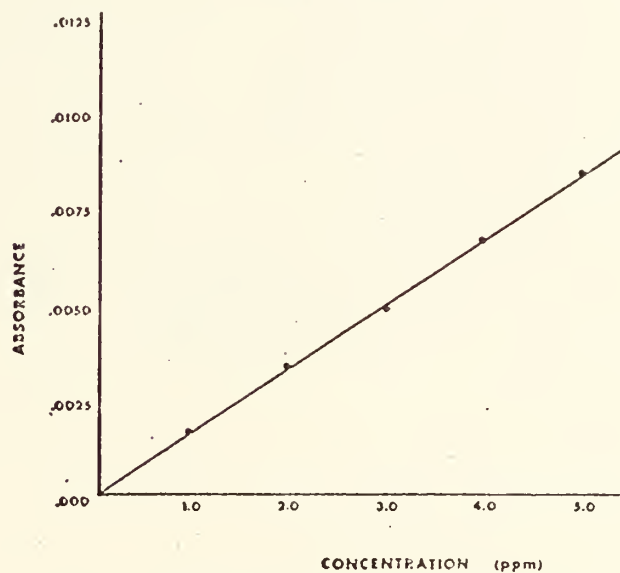
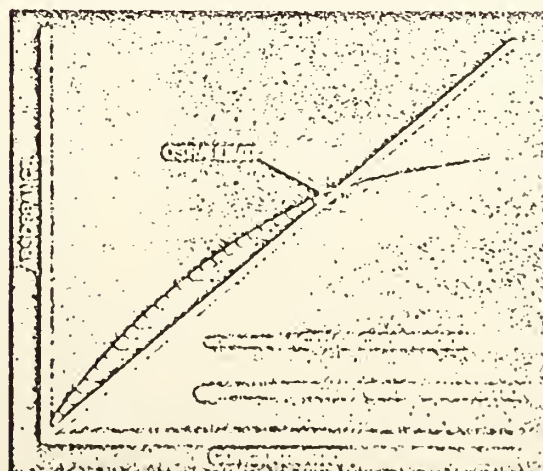


FIG. 3-3B



Calibration curve for low concentrations of vinyl  
chloride

FIG. 3-3C





APPENDIX B  
RECORD BOOK



APPENDIX C  
PATH OF AN H-3 BLADE THROUGH  
HELICOPTER BLADE BONDING SHOP





4348 (1000) 200 7/23/70



1001 1002 1003 1004

113 3247 1099 10.99

10.20.1

4343.2

113

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Figure 10-10

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1. *Chlorophyll a* (Chl *a*)

51861 *Willd.*

FINISH 4100

— 62 —

STAFF: 1992

100

1. The first step is to identify the problem or question that needs to be answered.

Received 2002.11.25

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(4)

1911

H. H. C. SUPER 2001

light hand roll pressure against pocket trailing edge. Adjust pivoting pocket flaps around head roll. Check alignment of

in head and wings using color coded pages.

3. Layers of aluminum foil tape, #425, #417 or aluminum foil, 3 mils, 1/8" on inside of heaters, 1/4" on 1 inch wide, 1/4" on 1/2 inch wide, 1/4" on 1/4 inch wide

1. Join ends of bond lines. Between adjacent and a minimum of 1/2" wide, over bond in a lot of adjacent pockets. - Install leaf sink in end leathers.

14 ea. clamps over beaters, equally spaced at each pocket  
clamps are utilized.

Controlled at 200°F. Cure 16: 1 hour and 30 minutes, power  
down of

START FINE

## FINISH FINE

test specimen, set controller at 255°F. Cure for 1 hour  
at 15 minutes, power on to power off.

blade to cool to 180° F before removing clamps.

pocket alignment

neck.

For final acceptance of bond.

Acceptance of bond.	QA.	DATE COMPLETED

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6,4 #2882.

. U. ORSINGER

1.2/10/75

121 V. 1. 12 053

Page 4 of 5





## APPENDIX D

### QUICK AND ROUGH PROCEDURES FOR HCN VAPOR CONCENTRATION DETERMINATION



AROMIL CHEMICAL COMPANY

P. O. Box 7627

Baltimore , Maryland  
21207

INSTRUCTIONS FOR THE USE OF AROMIL DETECTOR CRAYONS

Select the proper detector crayon or crayon pair for the gas that is to be detected. Make a detector crayon mark by rubbing the crayon back and forth on any suitable surface, such as a piece of paper, wood, hood wall, etc. This detector crayon mark will change color in the presence of the specified gas.

IMPORTANT! See the specific instructions for each crayon.

Phosgene Detector Crayon 1: The marks from this crayon will turn pink or possibly a salmon color in the presence of low concentrations of phosgene vapors. This mark will detect 1 p. p. m. of phosgene in about 30 to 60 seconds. Higher concentrations of phosgene will give an even more rapid color change. CAUTION: Very high concentrations of phosgene may bleach this pink mark and give a lighter color! Phosgene Detector Crayon 2 should be used in the presence of high concentrations of phosgene. For maximum safety and effectiveness, the use of side by side marks of Phosgene Detector Crayons 1 and 2 is recommended when testing for phosgene.

Phosgene Detector Crayon 2: This crayon is especially recommended for testing for leaks in an atmosphere containing traces of phosgene. In order to obtain the best results, the marks from this crayon must be a uniformly tan to light brown color before exposure to the phosgene. This color is obtained by rubbing the crayon over the writing surface such that the red coating becomes uniformly distributed throughout the mark. The resulting tan mark will turn green when exposed to high concentrations of phosgene. This green mark will gradually turn to blue and then to violet on standing. CAUTION: This crayon will NOT detect low concentrations of phosgene! Phosgene Detector Crayon 1 should be used for this purpose.

Phosgene Detector Crayon 2 may also be used to detect the presence of lewisite. The tan crayon mark will turn greenish-blue on contact with lewisite liquid or vapors.

Hydrogen Cyanide Detector Crayons A and B: Make a mark with Hydrogen Cyanide Detector Crayon A. Rub Hydrogen Cyanide Detector Crayon B over this mark to give a mixed mark which contains ingredients from the two crayons. This mixed mark will turn a reddish-pink in the presence of hydrogen cyanide. This mark will detect 10 p. p. m. of hydrogen cyanide in about five minutes. Higher concentrations of gas will give a more rapid color change. On standing, the pink color will darken to purple or blue.

Hydrogen Cyanide Detector Crayon B may be used by itself to detect cyanogen chloride or cyanogen bromide. The crayon mark will turn reddish-pink in about one minute when exposed to 4 p. p. m. of cyanogen halide vapors. Higher concentrations of gas will produce a more rapid color change, in proportion to the gas concentration.

NOTICE

These crayons will deteriorate slowly with time. It is recommended that, when not actually in use, these crayons be kept in their closed containers away from excessive heat. Because of this gradual deterioration, it is recommended that these crayons be discarded after six months.

Due to the widely varying conditions under which these crayons may be used, the user should determine for himself if these crayons are applicable to his particular needs.





# AROMIL CHEMICAL COMPANY

DIVISION OF SYNOrganics, INC.

P.O. BOX 7627

November 2, 1976

BALTIMORE, MD. 21207

Dr. John H. Duffin  
1014 F Avenue  
Corona, CA 92118

Dear Dr. Duffin:

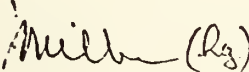
We refer to your ~~Purchase~~~~Order~~~~Max~~ inquiry.

We currently offer sensitive Phosgene and HCN Detector Crayons according to the following pricing schedule:

Phosgene Detector Crayon No. 1 .....	\$ 10.00 each
Phosgene Detector Crayon No. 2 .....	6.00 each
HCN Detector Crayons No. A & B .....	12.00 pair

We await receipt of your approval to ship your order under the current pricing schedule.

Sincerely yours,

  
J. I. Miller, Sales

JIM:hg

Telephone: 301-655-3081



# HAZARDOUS GAS DETECTOR

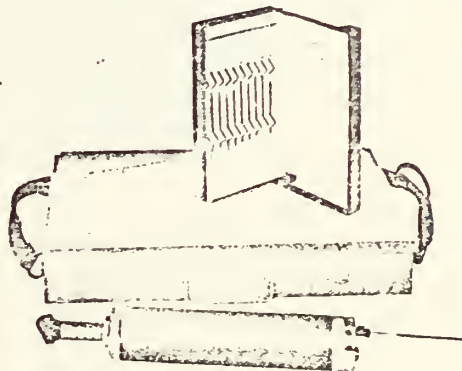
## MODEL 8014

The Model 8014 (Kitagawa) Hazardous Gas Detector provides accurate, dependable and reproducible results in determining concentrations of toxic gases and vapors. It has been proven through extensive use by leading industrial companies and government agencies.

Only three easy steps are required to operate the detector. (1) break off the tips of a fresh tube, (2) insert the tube end marked with a red dot into the pump inlet, and (3) pull out the pump handle to draw in a 100 cc. sample of atmosphere. These samples are drawn into the pump at a fixed and reproducible rate through a single, stainless steel, limiting micro-orifice. This single precision orifice and the design of the detector tubes eliminate the need for multiple orifices. The detector tube contains a chemical reagent which absorbs and reacts to the gas or vapor being analyzed. The tubes are formulated with a constant reagent weight which is corrected by a calibration chart for variations in each tube's diameter. All tubes are ready for use and are supplied with instructions and temperature corrections. In most cases a constant color stain is produced which varies in length according to the concentration of the material being measured. The length of the stain is measured against a calibration chart furnished with each box. For carbon monoxide (8014-106A, 106B, 106C) and ethylene (8014-108B) measurements the concentration is determined

by a change in color. A color chart is included with each box of tubes.

The pump has a solid brass, hand-honed, corrosion-proof piston. The handle is accurately calibrated and marked from 0 to 100 cc. to assure reproducible results. The pump is virtually maintenance-free; it requires no lubrication and carries an unconditional 5 year guarantee.



### PRICE

Model 8014 Precision, Hazardous Gas Detector, with pump, metal carrying case, spare parts, micro-photograph of pump micro-orifice, and operating manual. Without tubes (select tubes from chart)

**\$90.00**

Engineering Report with additional details available on request.





# HAZARDOUS GAS DETECTOR

TUBE No.	DETECTOR TUBE	MEASURABLE CONCENTRATION	SHELF LIFE	INTERFERENCES	PACKAGING Tubes Per Box	PRICE
8014-102A	Acetone	0.05-5.0%	unlimited	Organic vapors	10	\$7.50
8014-102C	Acetone - in presence of other hydrocarbons	0.01-4.0%	1 year†	Acetaldehyde, acrolein, methyl ethyl ketone, methyl isobutyl, ketone, SO <sub>2</sub>	10	8.00
8014-101	Acetylene	50-1,000 PPM	unlimited	Organic vapors	10	7.50
8014-105A	Ammonia - high range	1-25%	unlimited	Hydrogen Sulfide	10	7.50
8014-105B	Ammonia - low range	20-700 PPM	1 year†	Amines	10	9.00
8014-140	Arsine	5-160 PPM	unlimited	Hydrogen sulfide, phosphine	10	7.50
8014-118A	Benzene	10-310 PPM	1 year†	Toluene, xylene, hexane	10	8.50
8014-118B	Benzene - in the presence of other aromatic hydrocarbons	25-345 PPM	1 year†	none	10	10.00
8014-138	Butyl Acetate	0.01-1.0%	unlimited	Other organic vapors except halogenated hydrocarbons	10	7.00
8014-126A	Carbon Dioxide - high range	0.1-2.6%	unlimited	Acid gases at high concentration	10	9.00
8014-126B	Carbon Dioxide - low range	300-7,000 PPM	unlimited	Acid gases at high concentration	10	9.50
8014-106A	Carbon Monoxide	25-6,000 PPM	unlimited	Hydrocarbons, hydrogen sulfide, nitrous gases, ammonia	10	7.00
8014-106B	Carbon Monoxide - in the presence of ethylene	25-6,000 PPM	unlimited	Nitrogen dioxide	10	8.50
8014-106C	Carbon Monoxide - in the presence of hydrocarbons and nitrous gases	25-6,000 PPM	1 year†	none	10	8.50
8014-109	Chlorine	1-40 PPM	1 year†	Other halogens, ozone, nitrous gases	10	7.50
8014-116	Chlorine Dioxide	10-500 PPM	1 year†	Halogens, ozone, nitrous gases	10	9.00
8014-123	Dimethyl Ether	0.01-1.2%	unlimited	Organic vapors	10	7.00
8014-104A	Ethanol	0.04-5.0%	unlimited	Organic vapors	10	7.00
8014-111	Ethyl Acetate	0.01-5.0%	unlimited	Organic vapors	10	7.50
8014-108B	Ethylene	0.5-100 PPM	unlimited	Carbon monoxide, acetylene, hydrogen sulfide, hydrogen cyanide	10	8.50
8014-122	Ethylene Oxide	0.01-3.5%	unlimited	Organic vapors	10	7.50
8014-113	n Hexane	0.01-0.6%	unlimited	Organic vapors	10	8.00
8014-112A	Hydrogen Cyanide	0.01-3.0%	1 year†	Cyanogen, hydrogen sulfide, sulfur dioxide	10	7.50
8014-167	Hydrogen Selenide	1-600 PPM	1 year†	Arsine, hydrogen sulfide	10	7.50
8014-120A	Hydrogen Sulfide - high range	0.01-0.17%	unlimited	Sulfur dioxide	10	7.50
8014-120B	Hydrogen Sulfide - low range	5-160 PPM	1 year†	Sulfur dioxide	10	8.50
8014-120C	Hydrogen Sulfide - in the presence of sulfur dioxide	0.005-0.16%	unlimited	Hydrocarbons, carbon monoxide, nitrous gases, hydrogen cyanide	10	8.50
8014-120HH	Hydrogen Sulfide	1000-3000 PPM	unlimited	Sulfur dioxide	10	8.50
8014-153	Isobutyl Acetate	0.01-1.4%	unlimited	Organic vapors	10	8.00
8014-149	Isopropyl Acetate	0.01-1.2%	unlimited	Organic vapors	10	8.00
8014-150	Isopropyl Alcohol	0.01-2.0%	unlimited	Organic vapors	10	7.00
8014-157	Methyl Bromide	10-500 PPM	1 year†	Chlorine, bromine, hydrogen chloride, ethyl bromide	20	12.00
8014-139B	Methyl Ethyl Ketone	0.01-1.4%	unlimited	Organic vapors	10	7.00
8014-155	Methyl Isobutyl Ketone	0.005-1.0%	unlimited	Organic vapors	10	8.00
8014-164	Methyl Mercaptan	5-120 PPM	1 year†	Methyl sulfide, chlorine, acetylene, nitrogen dioxide, carbon monoxide, ethylene, ethyl mercaptan	10	9.00
8014-129	Nickel Carbonyl	20-700 PPM	1 year†	Hydrogen sulfide, sulfur dioxide	10	8.00
8014-117	Nitrogen Dioxide	1-1,000 PPM	1 year†	Halogens, ozone, chlorine dioxide	10	8.00
8014-135	Perchloroethylene	10-400 PPM	1 year†	Bromine, chlorine, nitrogen dioxide, methyl bromide, hydrogen chloride, trichloroethylene, 1,2-dichloro-ethylene	10	11.00
8014-146	Phosgene	.05-50 PPM	1 year†	Halogens, nitrogen dioxide, hydrogen chloride	10	8.00
8014-121A	Phosphine - high range	20-800 PPM	unlimited	Hydrogen sulfide, arsine	10	7.00
8014-121B	Phosphine - low range	5-90 PPM	unlimited	Hydrogen sulfide, arsine	10	7.00
8014-103A	Sulfur Dioxide - high range	0.1-4.0%	unlimited	Hydrogen sulfide	10	8.00
8014-103B	Sulfur Dioxide - middle range	0.02-0.3%	unlimited	Hydrogen sulfide	10	8.00
8014-103C	Sulfur Dioxide - low range	5-300 PPM	1 year†	Hydrogen sulfide	10	8.50
8014-103D	Sulfur Dioxide - trace range	1-80 PPM	1 year†	Hydrogen sulfide, nitrogen dioxide	10	8.50
8014-103BF	Sulfur Dioxide - in flue gases	0.02-0.30%	unlimited	none	20	12.00
8014-124	Toluene	1-1,000 PPM	1 year†	Benzene, xylene, hexane	10	9.50
8014-134	Trichloroethylene Perchloroethylene	10-400 PPM	1 year†	Halogens, chlorine, ozone, nitrous gases	20	12.00
8014-132	Vinyl Chloride	0.05-1.0%	unlimited	Organic vapors	10	8.00
8014-143	Xylene	50-5,000 PPM	1 year†	Benzene, toluene, hexane	10	8.00

† All short shelf life tubes can be extended by refrigeration.

\* Refrigeration recommended.





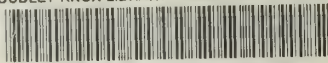
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